PB82-119082

Physical and Chemical Characteristics of Synthetic Asphalt Produced from Liquefaction of Sewage Sludge

Honolulu City and County Dept. of Public Works, HI

Prepared for

Municipal Environmental Research Lab.
Cincinnati, OH

Oct 81

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EPA-600/2-81-242
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PHYSICAL AND CHEMICAL CHARACTERISTICS OF SYNTHETIC ASPHALT PRODUCED FROM LIQUEFACTION OF SEWAGE SLUDGE

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TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO.	2.	3. RECIPIENT'S ACCESSION NO.
EPA-600/2-81- 242	ORD Report	PB82 119082

4. TITLE AND SUBTITLE
Physical and Chemical Characteristics of Synthetic Asphalt Produced from Liquefaction of Sewage Sludge

5. REPORT DATE
October 1981

6. PERFORMING ORGANIZATION CODE

7. AUTHOR(S)
J. M. Donovan, T. R. Batter, R. K. Miller, R. P. Lottman

8. PERFORMING ORGANIZATION REPORT NO.

9. PERFORMING ORGANIZATION NAME AND ADDRESS
Battelle Memorial Institute
Pacific Northwest Laboratories
P. O. Box 999
Richland, Washington 99352

10. PROGRAM ELEMENT NO.
DUB-113 PE-A7B1B Task B-23

11. CONTRACT/GRANT NO.
R-806790-01

12. SPONSORING AGENCY NAME AND ADDRESS
Municipal Environmental Research Laboratory - Cin., OH
Office of Research and Development
U. S. Environmental Protection Agency
Cincinnati, Ohio 45268

13. TYPE OF REPORT AND PERIOD COVERED
Final, Sept. 1979-April 1981

14. SPONSORING AGENCY CODE
EPA/600/14

15. SUPPLEMENTARY NOTES
Project Officer, Howard Wall (513)684-7659

16. ABSTRACT

Direct thermochemical liquefaction of primary undigested municipal sewage sludge was carried out to produce a low molecular weight steam-volatile oil, a high molecular weight synthetic asphalt, and a residual char cake. The latter product is capable of supplying the thermal energy requirements of the conversion process. The steam-volatile oil has immediate value as a synthetic fuel oil. The synthetic asphalt may prove to be a useful cement for paving with further research, or can be used as a fuel or coking stock. It is outwardly similar to petroleum asphalt, but chemically different.

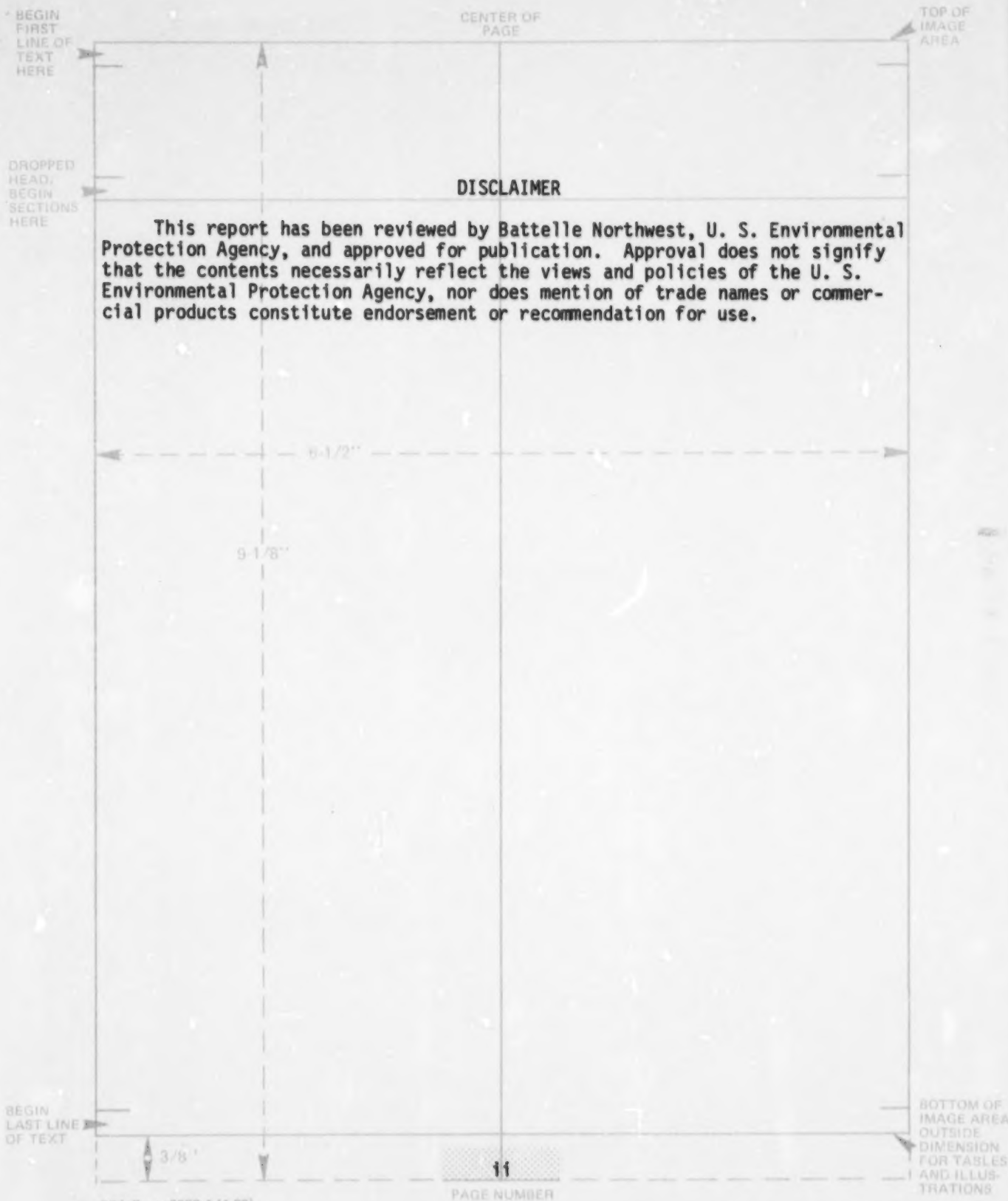
The thermochemical liquefaction process should be capable of operating technically and in an environmentally acceptable manner in conjunction with many existing waste-water treatment facilities. The overall feasibility of the process depends on the value of the oil and synthetic asphalt products as petroleum replacements, and on the costs associated with disposal of sludge. Projected economics indicate that the process has considerable promise for many potential sites in the United States at the present time.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
		13-B

18. DISTRIBUTION STATEMENT	19. SECURITY CLASS *(This Report)*	21. NO. OF PAGES
Release to Public	Unclassified	67
	20. SECURITY CLASS *(This page)*	22. PRICE
	Unclassified	

EPA Form 2220-1 (9-73)



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FOREWORD

The Environmental Protection Agency was created because of increasing public and government concern about the dangers of pollution to the health and welfare of the American people. Noxious air, foul water, and spoiled land are tragic testimony to the deterioration of our natural environment. the complexity of that environment and the interplay between its components require a concentrated and integrated attack on the problem.

Research and development is that necessary first step in problem solution and it involves defining the problem, measuring its impact, and searching for solutions. The Municipal Environmental Research Laboratory develops new and improved technology and systems for the prevention, treatment, and management of wastewater and solid and hazardous waste pollutant discharges from municipal and community sources for the preservation and treatment of public drinking water supplies, and to minimize the adverse economic, social, health and aesthetic effects of pollution. This publication is one of the products of that research, - a most vital communications link between the researcher and the user community.

The report describes the development of a process which will convert municipal wastewater sludge into a useful non-polluting product. The product is also a projection of a method that can be used to recover energy from wastes and save fossil fuels. This work is one of the tools necessary to assess how the ultimate disposal of municipal wastewater sludge can be done in a manner that will make it a truly valuable and prehaps profitable resource.

Francis T. Mayo
Director
Municipal Environmental Research Laboratory

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The thermochemical liquefaction process should be capable of operating technically and in an environmentally acceptable manner in conjunction with many existing wastewater treatment facilities. The overall feasibility of the process depends on the value of the oil and synthetic asphalt products as petroleum replacements, and on the costs associated with disposal of sludge. Projected economics indicate that the process has considerable promise for many potential sites in the United States at the present time.

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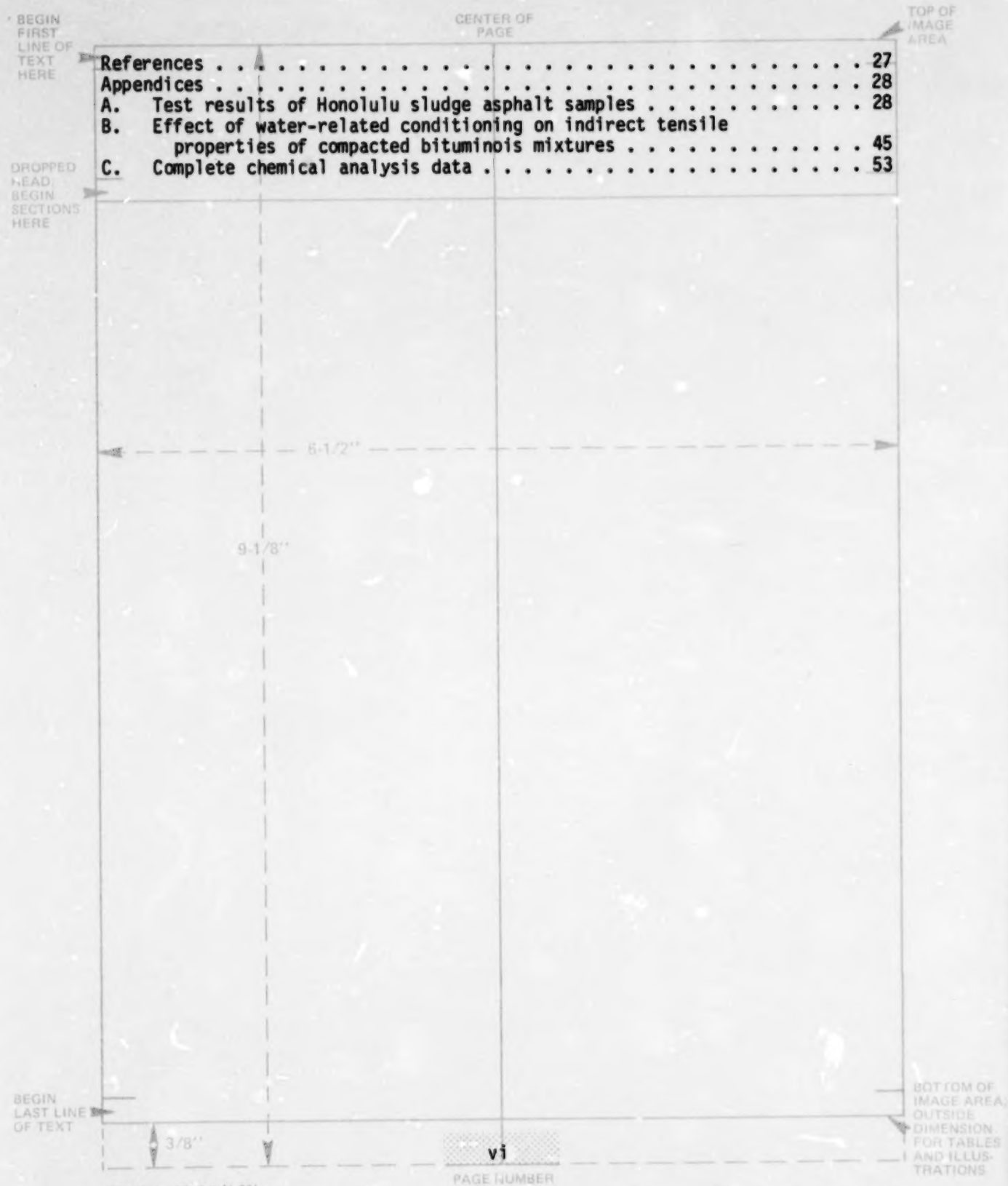

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INTRODUCTION

Disposal of sewage sludge is an increasing problem for many municipalities in the United States. There is currently a need for implementation of alternative disposal technologies. At the same time, it is recognized that alternative technologies need to be energy efficient, and that if possible some product of value should be recovered from sludge. Direct thermochemical liquefaction has the capability of meeting all of these requirements.

Thermochemical liquefaction investigations by various authors have shown that organic biomass can be converted to bitumen (1), heavy oil (2), and distillate oils (3) with combustion heats closely approximating petroleum products (4). An organic solvent liquefaction process also appears capable of producing asphalt-like material from various biomass forms (5).

The utility of the high molecular weight fraction of the liquefaction product (because it is a substantial part of the liquefaction products) is often questioned. To this end, the present study was conducted to determine the value of the high molecular weight fraction as synthetic asphalt for use as a paving cement. A synthetic asphalt product would be valuable since petroleum asphalt has increased in value with escalating petroleum costs and has become a target for increased utilization (e.g. production of olefins) as a petrochemical feed stock.

The low molecular weight product of biomass liquefaction has always been viewed as a valuable synthetic fuel. In the case of sludge liquefaction, the low molecular weight oil product was conveniently separated during the reaction period by steam auto-distillation. This steam-volatile oil was obtained in significant quantities using a relatively simple process. This process also led to simplified separation of excess water present due to the use of wet sludge.

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CONCLUSIONS

Seventy percent of the combustion energy available in sewage sludge (approximately 5000 cal/g or 9000 Btu/lb) can be converted to liquid and solid fuels analogous to petroleum by direct thermochemical liquefaction.* The solid fuel can be burned to provide all the necessary process heat requirements so that the process is a net energy producer.

The high molecular weight product of liquefaction is, in some cases, an acceptable replacement for petroleum asphalt based on the results obtained so far. Synthetic asphalt samples designated HS-1, 7, and 10 were ranked as satisfactory by our preliminary asphalt testing procedures, while most others were ranked as unsatisfactory. Since the conversion procedure for the satisfactory and unsatisfactory samples was the same in most cases, the only plausible explanation is that inherent differences in the composition of the various samples of sludge used led to physical differences in the synthetic asphalt products.

The synthetic asphalt can also be used as a potential fuel or coking stock in the event that it is not immediately acceptable as a paving binder. In addition, the steam-volatile oil produced during liquefaction has significant value as a synfuel, having 90% of the heating value of No. 2 fuel oil. Based on the current value of petroleum asphalt and heating oils, the liquefaction process would be economically viable in many existing situations, with expected payback periods of less than 12 years.

The extraction step required to produce an asphalt adds to process complexity and cost. Economics would be improved if the process objective was solely to produce fuels.

---

* In a representative experiment (HS-10) 1 kg. (dry basis) of undigested primary sludge having an approximate heat of combustion of 20.9×10^6 J was converted to 0.66 kg of liquid and solid products with combined heats of combustion of 14.9×10^6 J, or 71% of the original energy available in the sludge. The fuel requirement to process 1 kg of sludge is estimated to be 5.1×10^6 J (assuming that the fuel is burned at 80% efficiency). If the process is fueled by its own products, the net recovery of energy from undigested sludge is 14.9×10^6 J minus 5.1×10^6 J, or 9.8×10^6 J, which is equal to 46% of the energy originally available in the sludge.

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RECOMMENDATIONS

The potential for direct liquefaction of sludge to produce liquid fuel at reasonable cost is very real based on the data presented in this report. With this prospect, investigations should continue at full speed, since no other alternative shows as much promise except perhaps incineration. However, liquefaction produces a liquid fuel which can be stored or transported, rather than steam which must be used at or near the generation site and for a limited number of applications such as electric power generation or direct heating uses. The quantity of net energy produced by liquefaction (i.e. steam-volatile oil and synthetic asphalt) is likely to be greater than the net energy produced by incineration (i.e. steam in excess of process requirements).

Batch reaction methods used for this study are not acceptable for design and scale-up for even a modest pilot plant. Therefore, a small, lab-scale, continuous reaction system is needed to:

1) Determine physical properties of intermediate products

2) Determine necessary designs for ancillary equipment

3) Obtain larger samples for more detailed testing

4) Determine liquefaction product characteristics as a function of temperature, time, alkali, and incoming sludge composition

5) Determine the technical feasibility of a continuous reactor for sludge liquefaction.

Reliable data for design, scale-up, and economic evaluation would result from a continuous bench unit. In addition, if this unit were located at the site of a wastewater treatment plant, product characteristics as a function of sludge composition could be measured and the treatability of the residual aqueous phase could be determined.

Therefore it is recommended that a small lab-scale continuous liquefaction facility be built near a wastewater treatment plant, With this system, emphasis should be placed on development of fuel oil from sludge because this option appears to be more cost-effective in the near term. However, since the high molecular weight fraction is made along with the steam-volatile oil, investigations of its utility as a petroleum asphalt replacement or as a coking stock or fuel can continue at minimum additional R&D cost.

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THERMOCHEMICAL LIQUEFACTION OF PRIMARY SEWAGE SLUDGE

Previous work in the area of direct thermochemical liquefaction of sewage sludge and other forms of biomass has resulted in the production of high molecular weight fractions which are similar in appearance to petroleum asphalt. A high molecular weight fraction derived from pure cellulose in the Battelle-Northwest Laboratory showed promise as a replacement or extender for petroleum asphalt. Due to the high cost of pure cellulose and other biomass forms, other alternative feed materials were suggested. Primary undigested sewage sludge, because of its low or negative cost, was a logical choice.

Experimental Procedure

The intended feed stock for the experimental work was dried primary sludge from Honolulu. However, because of inavailability of drying equipment, the actual feed stock was fresh primary sludge, dewatered with polymer. The fresh sludge was preserved by addition of chloroform, and later by freezing. It was then sent by air to our laboratory at Richland, Washington.

Fresh sludge received at our laboratory was prepared for reaction by adding a base (either Na_2CO_3 or CaO). This material was then loaded into an Inconel liner and blanketed with an inert gas (Argon). The liner was sealed and placed into an autoclave. Water was added between the liner and autoclave to assist in heat transfer and to balance the differential pressure across the liner which would result from heating to reaction temperature. Pressure inside the liner was controlled to equal the vapor pressure of water in the annular space, to prevent either implosion or explosion of the liner.

After being prepared in this manner, the autoclave was heated to reaction temperature, which required 2 to 3 hours. The autoclave was held at reaction temperature for one hour, and the cooling cycle required 4 to 6 hours. During the heating, reaction, and cooling periods, gas and steam were emitted from the liner. This was caused by excess partial pressure in the liner caused by gas generation which accompanies thermochemical liquefaction. The predominant gas formed and discharged was carbon dioxide. However, steam and steam-volatile oil was also discharged with the gas. These were condensed in a water trap and saved for later analysis. Hydrogen sulfide was monitored during the reactions; however, none was detected.

After cooling, the autoclave was opened, the liner removed and opened, and the product discharged. This product could be separated into an aqueous supernatant liquid and a char cake by either settling or centrifugation. The latter was more convenient in laboratory work and was used most often.

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The supernatant liquid was analyzed for its volatile constituents and was subjected to treatability analyses. The char cake contained the high molecular weight fraction of the liquefaction product which was destined to become synthetic asphalt. As a result of being intermixed with the char and ash, the high molecular weight fraction was not acceptable for use as a replacement or extender for petroleum asphalt since it would not melt when heated and would not mix with heated petroleum asphalt.

To overcome the problems caused by the presence of char and ash, the high molecular weight fraction was separated by solvent extraction. Previous liquefaction work, and specifically work with liquefaction products from cellulose, indicated that acetone was an acceptable solvent. Soxhlet extractors were used, and extraction times were 8 to 24 hours. The extracted char cake was crumbly after solvent removal. The high molecular weight liquefaction products were recovered by removal of solvent in a rotary evaporator under vacuum. The resulting material resembled heavy crude oil in physical appearance. In preliminary work, it was not acceptable for direct use as a petroleum asphalt replacement or extender because its viscosity was not high enough.

Removal of residual low molecular weight liquefaction products from the extracted product by vacuum distillation solved the low viscosity problem. In practice, only a very small amount of low molecular weight material was removed. However, this small amount made a pronounced difference in the viscosity of the product.

Synthetic asphalt samples prepared in this manner were sent to Dr. R. Lottman at the University of Idaho for testing and analysis as paving cements.

Results

Reaction conditions and resulting product yields are given in Table 1. Results for HS-1 and HS-2 do not appear because of inadequate data. The mean total yield (oil+asphalt) for conversions carried out at 320°C with Na_2CO_3 was 20.4% with a standard deviation of 7.1%. Total yield seems to increase with temperature since the yields at 295 and 345°C differ from the mean by 1.5 and 2.3 standard deviations respectively.

Several runs at 320°C were done to provide the University of Idaho with a large, consistent sample for a complete series of asphalt paving material testing. Unfortunately, this prevented us from collecting data at other thermal conditions, which in turn limits our ability to describe yield as a function of temperature.

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TABLE 1 REACTION CONDITIONS AND YIELDS

Experimental Designation	Dry Ash-Free Sludge, Kg	Steam Volatile Oil, gm	Synthetic Asphalt, gm	Total Yield* %	Reaction Temp. °C	5% by Weight
HS-3	2.61	330	268	23	320	Na_2CO_3
HS-4	2.52	480	440	37	345	Na_2CO_3
HS-5	2.41	-0-	250	10	295	Na_2CO_3
HS-6	3.99	450	330	20	320	Na_2CO_3
HS-7	2.01	-0-	310	15	320	Na_2CO_3
HS-8	2.28	250	50	13	320	Na_2CO_3
HS-9	2.49	450	430	35	320	CaO
HS-10	1.33	250	164	31	320	Na_2CO_3

* Weight of light oil and synthetic asphalt as percent of dry, ash-free sludge.

The pressure at which these reactions took place was approximately equal to the pressure of saturated steam at the temperature of reaction.

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Lime (CaO) is apparently an excellent liquefaction adjunct*, having given approximately equal yield compared to carbonate under the same experimental conditions. Unfortunately, asphalt testing results indicated that the one sample we produced using lime was an inferior product. Due to the inherent variations in using sludge as a raw material, perhaps additional trials with lime would give better results.

Several of the synthetic asphalt samples sent to the University of Idaho showed a tendency to leach soluble material into water, especially during hot water soaking. This soluble material may have been removed and carried by the solvent during solvent extraction, or alternatively, water soluble material may have been formed during the vacuum distillation step. Under conditions of vacuum distillation (10 to 50 mm Hg, 200 to 300°C) some of the material may be subject to thermocracking, which could lead to formation of water soluble material. During the latter experiments, the samples were washed with water, after solvent removal, and were not heated beyond 200°C during distillation. This significantly reduced the water leaching problem. Further work in this area should reduce leaching to acceptable levels.

Odor production during the liquefaction reaction and subsequent operations was noticed, but these odor problems are not thought to present significant obstacles to further development of this technology, or future implementation of the process on a large scale. The odor was believed to be caused by indoles.

The large production of steam-volatile oil in these experiments exceeded our original expectations. This oil has significant value as a synfuel, having 90% of the heating value of No. 2 heating oil.

*Previous liquefaction work has referred to alkaline adjuncts as catalysts. However, our previous work has indicated that the alkali is a reactant, and that the overall reaction is highly dependent on pH. Because of this, alkalis used for liquefaction are referred to as adjuncts in this report.

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EVALUATION OF SYNTHETIC ASPHALTS AS PAVING CEMENTS

Approximately 10 samples of varying amounts of Honolulu sewage sludge asphalt (SSA) were evaluated as an asphalt extender or as an asphalt substitute. Because of limited supply for this development phase due to the small size of experimental equipment, feasibility tests were run consisting of a developed "preliminary test" and a compacted mix test. The preliminary test was used as a screening test and was not quantitative. The mix test was used when a sample passed the preliminary test and when there were adequate sample amounts to evaluate. This test was a quantitative test that measured compactibility and moisture-climate susceptibility using highway engineering mechanical properties of tensile splitting strength and resilient modulus (stiffness) (See Appendix A for complete individual reports and Appendix B for test method description).

An AC-10 petroleum asphalt was used as a reference control for these tests. Mineral aggregate used was from northern Idaho, a crushed basaltic rock used in highway paving. Crushed basaltic rock is also used extensively in Hawaii for paving mixes, which is why it was chosen for these experiments.

Preliminary Test

The preliminary test consisted of:

1. Determining the approximate melting and liquid (pouring) temperatures of the sludge asphalt samples,
2. Odor detection,
3. Leaching and cleaning in hot water,
4. Determining brittleness, tackiness as a thin film exposed to freezing and to room temperature,
5. Mixing with petroleum asphalt (for a 50-50 blend),
6. Mixing, coating and adhesion with hot, mineral aggregate,
7. Determining effect of warm water on the SSA-aggregate mixtures, and
8. Determining the effect of water (vs. dry condition) on the bending pulling-ductility characteristics of the SSA-aggregate mixtures, after consolidation.

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Steps 6-8 above consisted of the evaluation of SSA as a complete asphalt replacement (100% SSA) and also as an asphalt extender (50% SSA and 50% petroleum asphalt by weight).

SSA samples (100% SSA or 50-50% SSA) which passed the preliminary test had to perform roughly equivalent to petroleum asphalt. Samples that did not pass had one or more of the following characteristics:

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a. They were brittle at room temperature.

b. They leached out impurities severely when the SSA-aggregate mixture was exposed to water.

c. They imparted crumbly and weak characteristics to the SSA-aggregate mixture.

Compacted Mix Test

The compacted mix test consisted of incorporating 100% SSA (and sometimes 50-50% SSA) at 6% by wt. of mixture (design binder content determined by petroleum asphalt control mixture) and at approximately 6.9% permeable mixture voids after kneading compaction. Mechanical properties of tensile splitting strength and resilient modulus were measured in dry, vacuum saturated and accelerated field conditions. The accelerated conditioning consisted of saturation in water while under vacuum followed by 16 hr. freezing and 24 hr. of 60°C (140°F) water soak. The magnitudes of strength and modulus were compared to the petroleum asphalt control mixture, and the percentage retained (dry basis) of the strength and modulus were also compared after vacuum saturation and accelerated conditioning. The objective was to determine if the SSA sample imparted mechanical properties and moisture-climate resistance approximately equal to the petroleum asphalt control mixture.

SSA-aggregate mixtures that "passed" the compacted mix test needed to have dry tensile strength properties around 345 kPa (50 psi) at 12.8°C (55°F) and dry modulus properties around 1,379,000 kPa (200,000 psi) at 22.2°C (72°F). Also, they needed to have at least 90% retained tensile strength and modulus after vacuum saturation and at least 70% retained tensile strength and modulus after accelerated conditioning.

SUMMARY OF TEST RESULTS AND PROPERTIES

Individual test reports on SSA samples are attached in Appendix A. Sample designation HS #1 was a "beginning sample" made from sludge from the Richland, Washington sewage plant. All other HS samples were made from sewage sludge from Honolulu, Hawaii.

Odor and Leaching of Impurities.

All SSA samples had a characteristic pungent odor and leached in water, but these characteristics varied in degree. Some of the odor seemed to be

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removed during SSA sample washing. Washing was used with samples HS #6, 7, 9 and 10 primarily to remove impurities so that minimum leaching would occur from the SSA after mixing with aggregate and exposure to water. In these cases the color of the leachate after aggregate mixing would be a light yellow (solution). (Unwashed samples produce a dark olive brown leachate with suspended "impurities"). In most of the cases, the sample washing to remove impurities improved the SSA by reducing the SSA's moisture sensitivity.

Consistency and Appearance.

The SSA samples were black in color, resembling petroleum asphalt. They bonded well and were tacky. Most had more putty-like characteristics than petroleum asphalt. They melted and became liquid at temperatures in the range of petroleum asphalt. Their consistencies varied at room temperature from soft to moderately hard and brittle. At freezing temperature they all were brittle, some not more brittle than petroleum asphalt. All samples (except HS #10) seemed to lose heat and change consistency more rapidly than petroleum asphalt indicating a lower specific heat for the SSA.

Mixing with Petroleum Asphalt.

The SSA samples appeared to mix with petroleum asphalt as an asphalt extender, but there were reactive tendencies: "gel" formation and weakened binder resulted with most blends. After completion of preliminary tests on HS #10, it was concluded that SSA blends with petroleum asphalt may be adverse. It may be due to either the amount of SSA in the blend to an undefined chemical incompatibility.

Binder Characteristics with Aggregate Mixtures.

The SSA samples mixed with and coated hot mineral aggregate. There was definite binder action and the appearance of the mixture looked similar to petroleum asphalt-aggregate paving mixtures. Near the end of the SSA sample testing, when washed SSA samples were used, the 100% "blends" of SSA performed better than the 50-50% blends of SSA and petroleum asphalt in these mixtures. The 50-50% blends usually produced weak, crumbly mixtures whereas the 100% blends produced ductile, stronger mixtures.

In the presence of water, most of the SSA samples were too water sensitive. They softened or leached badly, resulting in weaker mixtures. Exceptions were HS #1, #7 and #10 SSA samples; they held up relatively well as compared to the petroleum asphalt control-aggregate mixture.

Mechanical Properties of Mixtures.

The tensile splitting strength and resilient modulus of the SSA-aggregate compacted mixtures, when performed with adequate sample amount, covered a wide range. Normally if a sample appeared hard and brittle in the preliminary test, it also had very good mechanical properties. Sometimes these good mechanical properties were drastically reduced in the presence of water and climatic action from the accelerated conditioning.

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However, a relatively successful sample like HS #7 gave mechanical properties in the range of petroleum asphalt-aggregate paving mixtures.

CONCLUSIONS FOR SYNTHETIC ASPHALTS AS PAVING CEMENTS

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1. Binding properties are present in SSA samples. Their cohesive properties are tackiness, but they are more putty-like than petroleum asphalt.

2. SSA samples give off an indole odor and leach in the presence of water to various degrees. This can be minimized by washing the SSA samples with water, and this seems to result in improved binder properties.

3. SSA consistency varies from soft to hard. The sample can be brittle (like HS #9) or softer (like HS #7).

4. SSA samples can be made to have consistency and mechanical properties similar to petroleum asphalt.

6-1/2"

5. Most samples are water susceptible and, at the best, are average in this category as compared to petroleum asphalt. It appears that HS #1, #7 and #10 are the least water susceptible of the SSA samples tested.

9-1/8"

6. The chemical make-up of most of the SSA samples may be responsible for an adverse reaction with petroleum asphalt. At this stage SSA may be better as an asphalt substitute rather than as an asphalt extender until more is known about SSA chemistry.

7. SSA samples HS #1, #7 and #10 rank as the best HS samples test to date. The other HS samples tested were declared unsatisfactory.

RECOMMENDATIONS FOR SYNTHETIC ASPHALTS AS PAVING CEMENTS

Since there is promise that a satisfactory SSA samples can be made to replace petroleum asphalt, it is recommended that larger samples be made utilizing a consistent sludge source and the best techniques developed during this experimental work so that:

1. The SSA can be evaluated with a full sequence of tests used for paving asphalt and mixtures, including mix design, and

2. Pending the successful outcome of 1., above, design and set-up a pilot plant to produce an SSA for a road pavement section as part of a demonstration project.

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CHEMICAL CHARACTERIZATION OF LIQUEFACTION PRODUCTS

Six analytical methods were used to analyze the products of sewage sludge liquefaction. Steric exclusion chromatography was used to determine molecular weight distribution. The carbon (C), hydrogen (H), nitrogen (N), and oxygen (O) contents of the samples were determined by using an elemental analyzer. The concentrations of sulfur (S) as well as the concentrations of chromium (Cr), cobalt (Co), nickel (Ni), zinc (Zn), mercury (Hg), and lead (Pb) were determined by x-ray fluorescence spectroscopy. Neutron activation analysis gave the concentrations of cadmium (Cd). The most abundant volatile chemical components were identified by gas chromatography/mass spectroscopy (GC/MS). A bomb calorimeter was used to determine heats of combustion.

The analytical results are summarized below. Details for analyses which generated large amounts of data are shown in Appendix C.

Molecular Weight Distribution by Steric Exclusion Chromatography

Samples dissolved in tetrahydrofuran were analyzed using a Waters Associates high pressure liquid chromatography system. The system was calibrated by analyzing eight polystyrene and polyethylene glycol standards covering the molecular weight range from 200 to 8500. The chromatograms of five synthetic asphalt samples and one petroleum asphalt sample are shown in Figure 1. Each sample eluted in 10 to 21 minutes; these times are indicated by marks on the chromatograms. In steric exclusion chromatography the highest molecular weight compounds elute first. The high molecular weight compounds indicated by the petroleum asphalt chromatogram (Chevron AC-10) immediately after the 10 min mark are missing in the synthetic asphalt chromatograms. The lack of these high molecular weight compounds coupled with a greater abundance of low molecular weight products resulted in a weight-average molecular weight for the synthetic samples which is half the average for the petroleum sample. As noted in Figure 1, the weight-average molecular weight for the synthetic asphalt is close to 600 and for petroleum asphalt it is 1280. Among the synthetic samples, the traces for HS-6 and HS-10 have very similar shapes, which is not surprising since they were reacted under the same conditions (320°C with 5% Na_2CO_3). Reaction temperature had some effect on the shape of the chromatogram as indicated by HS-5 (reacted at 295°C) and HS-4 (reacted at 345°C). The chromatogram generated by sample HS-9 shows the most difference. HS-9 was reacted at 320°C but with 5% CaO adjunct.

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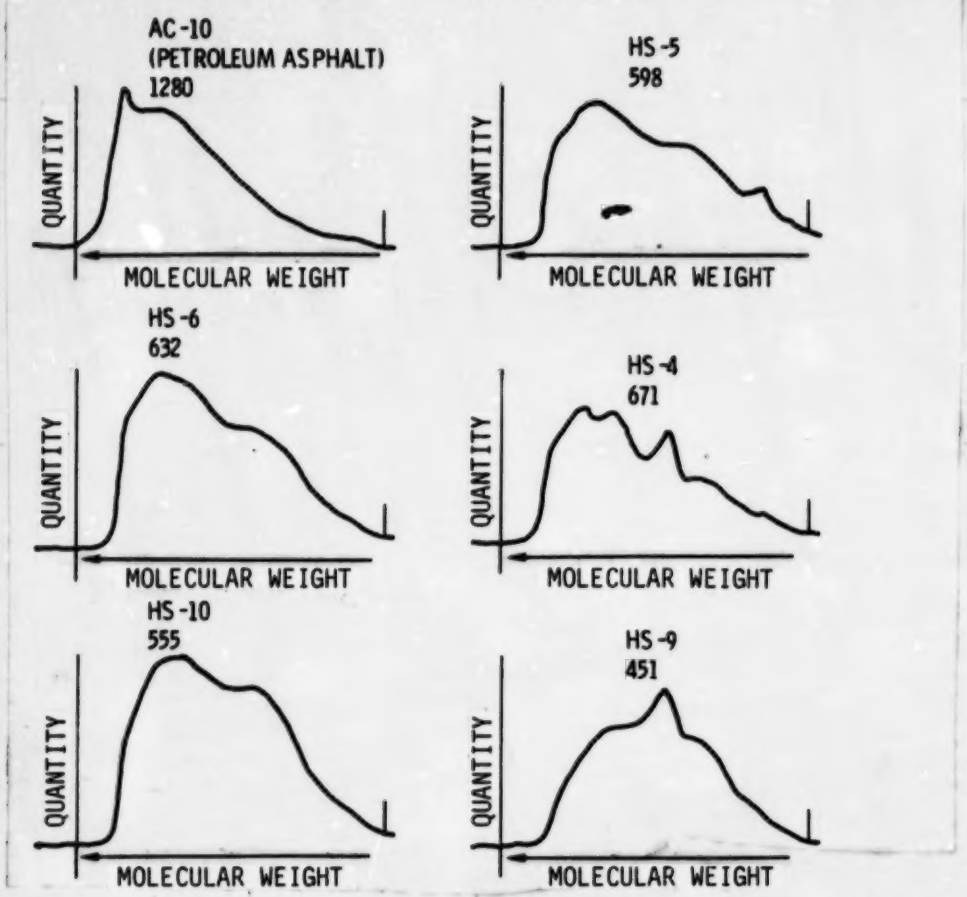

FIGURE 1. Steric Exclusion Chromatograms of Synthetic and Petroleum Asphalts

Although changes in molecular weight distribution occur with changes in reaction temperature and liquefaction adjunct, significant quantities of high molecular weight material were evident in the synthetic asphalt samples. Also, the evidence of low molecular weight material in the synthetic samples may have contributed to a problem noted earlier--water leaching. This fraction of the synthetic asphalt samples may have a low enough molecular weight to be significantly soluble in water.

Elemental Analysis

Table 2 lists percentages of C, H, N, O, and S in each of the types of liquefaction products and in petroleum asphalt. The values in the table for

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final products and for distillates are averages; the complete set of values is in Appendix C. Oxygen concentrations were measured directly by using an elemental analyzer. Sulfur values included in Table 3.1 are the total sulfur concentrations determined by x-ray fluorescence spectroscopy.

TABLE 2 ELEMENTAL ANALYSES

	Percent by Weight				
Element	Petroleum Asphalt AC-10	Synthetic* Asphalt	Steam-volatile** Oil	Leachate HS-6	Char Cake HS-10
C	87	74	77	59	26
H	11	10	12	9	3
N	0.4	4	3	3	0.9
O	1	9	7	18	10
S	-	0.8	0.9	0.9	-

* Average values from Experiments HS-4, HS-5, HS-6, HS-9, and HS-10.
** Average values from Experiments HS-9 and HS-10.

The reference to HS-6 leachate noted in Table 2 refers to material leached from HS-6 synthetic asphalt by hot water. The leachate was dried prior to analysis here and for subsequent analyses.

The sulfur concentration in the synthetic asphalt and oil samples reported in Table 2 would place them in approximately a grade four heating oil category. Both the sulfur and nitrogen contained in the synthetic asphalt samples and oils result from the use of sludge. Our analyses of volatiles (reported next) show that sulfur and nitrogen are substituted into the aromatic and aliphatic constituents of the volatile products. As a result of this, we suspect that the synthetic asphalt and oil products also contain a wide range of substituted sulfur and nitrogen compounds. For use as fuel, this is probably of minor concern. However, for use as synthetic asphalt, the presence of nitrogen may be limiting because of potential interactions between petroleum asphalt and the more polar synthetic asphalt (if synthetic asphalt is blended with petroleum asphalt). Also, since nitrogen is chemically substituted, and since the average molecular weight of synthetic asphalt is lower than petroleum, there may be a more pronounced tendency for synthetic asphalt to be soluble in water and for water solubility in synthetic asphalt to be higher than for petroleum asphalt.

Identification of volatile chemical constituents by GC/MS.

We analyzed three representative samples from two experiments:

- headspace of liquefied sludge (within 8 hours following liquefaction),
- headspace of liquefied sludge (several weeks after liquefaction),
- steam-volatile oil.

The compounds identified in five of the seven most abundant GC peaks for each sample are listed in Table 3.

TABLE 3 VOLATILE CONSTITUENTS

*Headspace 1	Headspace 2	Steam-volatile oil
Acetone	Acetone	Dimethylbenzene
Furan	Pentan-2-one	Ethylbenzene
2-methyl furan	2,3-dimethyldisulphide	p-cresol
Thiophene	Methyl thiophene	2-ethyl-1-hexanol
Toluene	Toluene	Ethyl indole

All of the compounds identified in the three samples are listed in Appendix C, organized first by sample and then by compound.

Trace Metal Analysis

The concentrations in ppm of seven trace metals are presented in Table 4.

Heavy metal concentrations reported in Table 4 are relatively low. We suspect that the values reported would be acceptable in most applications where synthetic asphalt is used as a paving binder and where the oil is used as a fuel.

The sludge used in these experiments came from primarily residential sources. Therefore, the trace metal concentrations may be relatively lower than would be expected from sludges with greater industrial waste contribution.

Heats of Combustion

The heats of combustion shown in Table 5 were determined on an ash-free basis except as noted.

* See page 54 for description of headspace.

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TABLE 4 TRACE METAL CONCENTRATIONS

	Parts per Million							
	Synthetic Asphalt					Steam-volatile		Leachate
Metal	HS-4	HS-5	HS-6	HS-9	HS-10	HS-9	HS-10	HS-6
Pb	3	3	9	11	8	<2	<2	4
Cd	<6	<6	<6	<6	<6	<6	<6	<6
Zn	32	27	52	24	18	16	40	29
Hg	<1	4	<1	<1	<1	9	2	<2
Cr	<3	10	4	<3	<3	<3	<3	5
Ni	100	64	15	18	12	2	1	12
Co	<3	<3	<3	<3	2	<1	<1	<3

TABLE 5 HEATS OF COMBUSTION

	Synthetic Asphalt		Steam-Volatile Oil		Leachate	Char Cake
	HS-9	HS-10	HS-9	HS-10	HS-6	HS-10
cal/g	8730	9060*	9380	9410	7760	3020*
Btu/lb	15,700	16,300*	16,900	16,900	14,000	5,400*

* Calculation based on total mass including ash.

The combustion heat of char cake shown in Table 5 appears low. However, this is due to the large concentration of ash in the char cake. The HS-10 char cake contained 60% ash prior to combustion.

Heats of combustion for the synthetic asphalt and oil reported in Table 5 are approximately 90% of the values for petroleum equivalents. Although not measured, the viscosity of steam-volatile oil was approximately that of No. 2 heating oil, judging from its pouring properties at room temperature.

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IDENTIFICATION AND BIODEGRADATION OF AQUEOUS PHASE ORGANIC COMPOUNDS

The aqueous phase from the sludge liquefaction process contains organic compounds. One possible method of treating these compounds is to recycle the aqueous phase back to the wastewater treatment plant where the sludge originated. Our evaluation indicates that at the levels of dilution we would expect in a typical plant, the recycle stream is not toxic to the flora of a typical sewage treatment plant and that a major portion of the organic compounds will be degraded by this flora.

Based on discussions with water treatment experts here at Battelle-Northwest, our actual experimental procedure differed in this task from the original proposal. We originally proposed to use culture of Pseudomonas aeruginosa and selected mixed cultures. We actually used seed cultures developed in our laboratory from raw sewage obtained from the Richland, Washington, sewage treatment plant. We developed these cultures using methods outlined in Standard Methods for the Examination of Water and Wastewater (6). We felt that the use of normal sewage treatment plant flora would give a better indication of the effect of the recycle stream on a sewage treatment plant. The gas chromatographic analysis of organic compounds in the solution is described in the chemical analysis section and therefore will not be repeated in this section of the report.

Initially, we determined the total organic carbon (TOC), the chemical oxygen demand (COD), and the biological oxygen demand (BOD). We used these values along with flow rate, BOD, and settleable solids data from a typical waste treatment plant to evaluate the effect of recycling the aqueous phase. We determined the TOC using a Beckman TOC Analyzer. We analyzed the COD by the Ampule COD digestion and analysis system developed by Oceanographic International Corporation, and approved by the EPA as an alternative COD procedure for nationwide use (7). This method uses sulfuric acid and potassium dichromate as oxidizing agents, and silver sulfate as a catalyst to chemically oxidize nearly all organic compounds. The COD values are determined by spectrophotometer analysis. The COD values determined by this method represent an estimate of the theoretical maximum BOD value. Based on this theoretical maximum value, we made appropriate dilutions, and determined the average BOD value using the method detailed in the Standard Methods for Examination of Water and Wastewater (6). As previously mentioned the seed culture for this BOD test was developed from raw sewage obtained from the City of Richland municipal sewage treatment plant.

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The average values of TOC, COD, and BOD we found by the previously described methods are as follows:

TOC	31.8 g/l
COD	93.4 g/l
BOD	38.9 g/l

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Figure 2 depicts the flow rates, settleable solids, and estimated BOD's of a sewage treatment plant and sludge liquefaction facility based on 10,000 liters of raw sewage per day. Based on the values shown in Figure 2 we estimate that the aqueous recycle stream will add an additional 2.9% BOD to the existing wastewater load. In addition, the COD and BOD values indicate that approximately 42% of the chemically oxidizeable compounds present in the aqueous stream were biologically degraded in the five day BOD test period. Finally, dilutions used to perform the BOD test were on the order of 1 part per 10,000. The aqueous phase was apparently not toxic to the sewage treatment flora at this concentration. Based on the flow rates shown in our typical facility in Figure 2, the aqueous stream would be diluted approximately 2.5 parts per 10,000. Since these dilutions are on the same order of magnitude, it is reasonable to expect that the aqueous phase recycled from the liquefaction plant will not be toxic to the sewage treatment flora at the expected dilution ratio.

Based on the preliminary results obtained here, we feel that the aqueous phase from the sludge liquefaction process can safely be recycled back to the sewage treatment plant without significant effect on the operation of this plant.

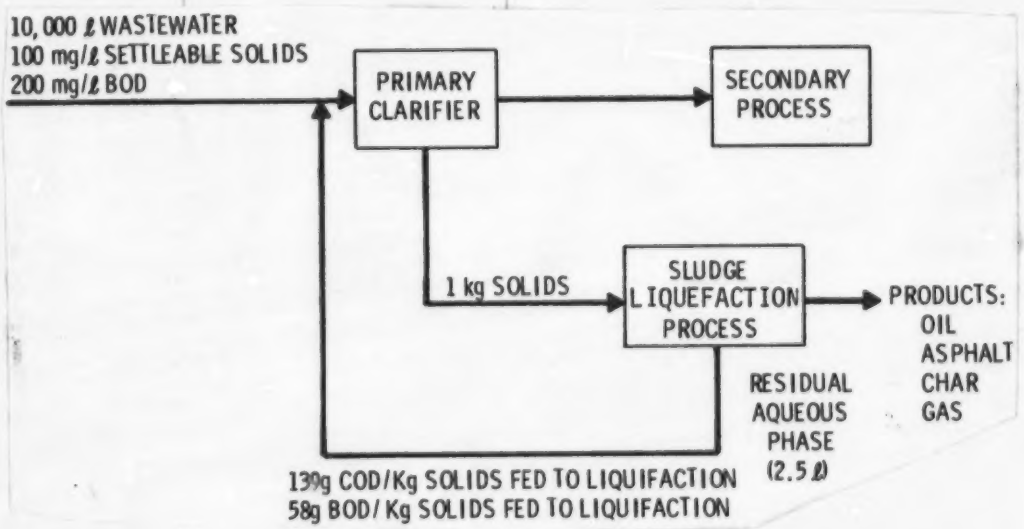

FIGURE 2. Recycle Scheme for the Residual Aqueous Phase

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CONCEPTUAL DESIGN AND COST ESTIMATES FOR A 91 TONNE/DAY* COMMERCIAL SLUDGE THERMOCHEMICAL LIQUEFACTION PLANT

The objective of this conceptual plant design and cost estimate is to help determine the overall feasibility of the process and to justify further research and development on continuous liquefaction of sewage sludge. Estimates have been made on the basis of the best available information at this time. These estimates are tentative since there is no pilot plant data, or even bench-scale continuous process data to support equipment specifications.

A flowsheet of the process is shown in Figure 3. Approximate product flows and temperatures are given for reference. A more detailed analysis of heat and mass balance is not currently warranted since heat of reaction, product yields, and processability of some of the streams in a continuous process is currently unknown.

Process Design

A plant capacity of 91 dry tonnes per day (100 short ton/day) was chosen as representative of the volume of primary sludge produced in large wastewater treatment plants around the United States. Therefore, the estimates given are for commercial plants and not for pilot or demonstration plants.

The process will operate on primary sludge which is dewatered to at least 30% solids. Other sludges can be used, but for these estimates, only primary sludge was considered. Dewatering to 30% solids is already practiced in many wastewater treatment plants. Many of these plants already use lime to condition the sludge for dewatering, adding in many cases 10% or more lime on a dry weight basis. In experiment HS-9 lime was demonstrated to be an effective adjunct for production of steam-volatile oil, but rated poorly for production of synthetic asphalt. Therefore, lime-treated sludge can be used directly without further treatment if the primary goal is fuel production. Synthetic asphalt from lime-treated sludge would require more investigation based on current results.

The thermochemical liquefaction yields given in the early part of this report showed significant variation. For the purposes of this design and estimation work, we have used the sludge characteristics and yields obtained from HS-10 as representative. Using this data, the yields from a plant processing 91 tonne/day would be:

*100 Short ton/day

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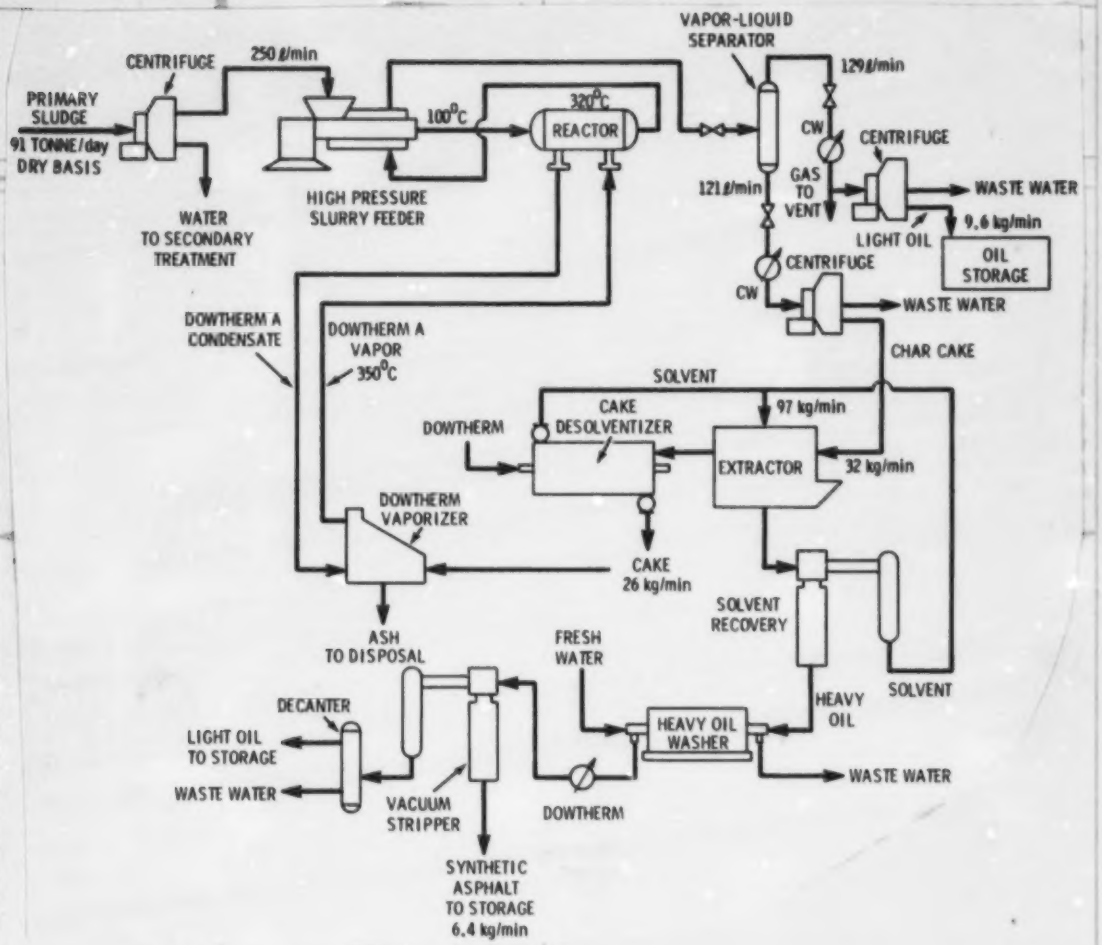

FIGURE 3. Preliminary Schematic for a Sludge Liquefaction Plant

Synthetic Asphalt: 9000 Kg/day
Steam-Volatile Oil: 13,800 Kg/day
Char Cake: 37,000 Kg/day

At a density of approximately 830 gm/l the light oil would amount to approximately 16,300 l, or just slightly greater than 100 petroleum barrels per day.

Process heat requirements for this plant will be supplied by combustion of the residual char cake. The extracted cake has a heat of combustion of approximately 3020 cal/gm. This cake is produced at the rate of 26 Kg/min and therefore is capable of supplying 1.56×10^{10} J/hr (1.5×10^{7} BTU/hr) of process heat at a combustion efficiency of 80%. This amount is more than adequate to supply process heat requirements, especially if heat recovery is used on the main stream coming out of the liquefaction reactor. The greatest

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heat requirement by far will be to heat the liquefaction reactor, even with heat recovery. In the case presented in Figure 3, the liquefaction reactor will require heat input of 1.17×10^{10} J/hr. Other minor heat requirements for solvent extraction and recycle are estimated to be less than 0.4×10^{10} J/hr. Total energy requirements therefore, are balanced by the available energy in the residual char cake. Energy from combustion of the char cake will be supplied to the liquefaction reactor by the Dowtherm vaporizer. Other process utility requirements are limited to electrical power of about 1.8×10^6 KWH/yr, and a small amount of cooling water.

Estimates given for capital equipment and manufacturing costs are derived from a plant which includes sludge dewatering equipment (a centrifuge) and solvent extraction to recover synthetic asphalt. Since many sites have dewatering equipment in place, and since some plants may elect not to recover synthetic asphalt, estimates for these processes will be given later in this report.

Equipment Description

The process design given in this section of the report is a conceptual design. This is necessary since the process is currently in the laboratory stage, and no adequate design data is available for a continuously operating plant. However, it is necessary to estimate actual plant costs to determine the potential commercial feasibility of the process and to provide justification for further research and process demonstration.

Table 6 includes all major pieces of equipment necessary to carry out the process along with preliminary cost estimates which were in most cases obtained in 1980 from the equipment manufacturers indicated.

Plant Capital and Manufacturing Costs

The total fixed capital investment required for a plant utilizing the equipment listed above would be in the range of $9.8 million. The total fixed capital investment (FCI) for the dewatering centrifuge is estimated at $2.0 million. The remainder of the plant is assumed to be analogous to conventional chemical processing industries, and as such the FCI for the remainder of the plant (equipment cost = $2.44 million) would be approximately $7.8 million. The total FCI for this plant including dewatering would be $9.8 million, as reported in Table 8.

Annual manufacturing costs are difficult to estimate at the present time. However, reasonable estimates of major manufacturing cost elements are presented in Table 7. These estimates assume that the manufacturing plant is part of a municipality which has the capability to use the products produced without significant freight, packaging, or selling expenses.

In addition to this, no cost is assumed for the raw material, sludge. This should be the case for many municipalities in the United States. In fact, many municipalities which face expensive disposal options may credit

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TABLE 6 MAJOR EQUIPMENT

Item	Capacity	$000 Cost Estimate	Function	Representative Manufacturer
Sludge dewatering centrifuge	1670 l/Min	1500	Concentrate primary sludge to 30% solids	Dorr-Oliver
Extruder	250 l/Min	250	Feed sludge into reactor	Berstorff
Reactor vessel	250 l/Min	350	Convert sludge to oil and synthetic asphalt	Manning & Lewis
Heat exchangers	Various	235	Miscellaneous plant heating and cooling functions	Manning & Lewis
Let-down valve system	250 l/min	100	Reduce product pressure to atmospheric	
Oil-water centrifuge	129 l/Min	90	Separate light oil from water in distillate	Dorr-Oliver
Char-water centrifuge	121 l/Min	90	Separate char cake from residual process water	Dorr-Oliver
Solvent extractor	32 Kg/Min	158	Extract synthetic asphalt from char cake	French Oil Mach.
Cake solvent dryer	32 Kg/Min	125	Recover solvent from char cake	Bepex
Synthetic asphalt washer	6.4 Kg/Min	60	Remove water solubles from asphalt	Baker-Perkins
Solvent stripper	19 Kg/Min	32	Recover solvent from synthetic asphalt	Artisan Indus.

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TABLE 6 (Continued)

Item	Capacity	$ 000 Cost Estimate	Function	Representative Manufacturer
Vacuum stripper	6.4 Kg/Min	32	Strip residual water and oil from synthetic asphalt	Artisan Indus.
Liquid decanter	0.5 Kg/Min	10	Separate water and oil resulting from vacuum stripping	Artisan Indus.
Vapor-liquid separator	250 /Min	60	Separate vapor and liquid products from reactor	Artisan Indus.
Dowtherm process heating system	1.56 X 10^{10} J/hr	400	Provide process heat	Foster-Wheeler
Storage tanks	various	450	Provide storage for oil and asphalt products, and char cake	Kennedy Tank & Mfg.
Total		3,942		

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TABLE 7 ANNUAL MANUFACTURING COSTS

Materials	none, credit taken for sludge disposal
Solvent replacement	$ 100,000
Management (with overhead)	90,000
Operating labor (with overhead)	400,000
Maintenance and supplies	630,000
Utilities	100,000
Fixed charges (including depreciation)	1,250,000
Ash disposal	400,000
Total	$2,970,000

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the liquefaction plant with part or all of the cost of sludge disposal (note that the manufacturing cost estimate includes an expense for ash disposal from the liquefaction plant).

As with the equipment estimate, the manufacturing costs are sufficiently conservative to allow for changes as a result of further process development. In the next section, four different plant options are presented with accompanying fixed capital investment and manufacturing cost requirements. These different options demonstrate the economics of the process depending on whether synthetic asphalt is recovered, and depending on whether the chosen site already has sludge dewatering equipment available.

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Simple Payback Period For Different Process Options

A major cost in this process is associated with primary sludge dewatering. Estimates given in Table 8 show that if investment for this process is not required, the pay back period for the plant will decrease from 10-12 years to 6-7 years.

Synthetic asphalt may be a valuable product when produced from sewage sludge. However, the estimates given in Table 8 show that the payback period is actually shorter if asphalt is not recovered. This result is caused by the solvent extraction process, which adds more capital and operating cost than can currently be recovered by sale of synthetic asphalt. If synthetic asphalt were not recovered, it would be contained in the char cake and simply burned. The viability of synthetic asphalt recovery will change depending on the yield of synthetic asphalt (as yet to be determined in a continuous process for which these estimates were made), and depending on its value as an alternative to petroleum asphalt. Either or both of these factors could significantly change the economics of synthetic asphalt recovery from sludge in future years.

In Table 8 credit for sludge disposal was estimated at $66 per dry tonne ($60/short ton). Revenue from sale of oil and synthetic products was calculated at $0.25/ ($40/bbl) for the light oil and $143/tonne ($130/short ton) for the synthetic asphalt, which are representative of 1980 prices for equivalent petroleum products.

At current petroleum prices, and with the capital and operating costs shown, it is necessary to take some credit for sludge disposal in order to make the process economically viable. However, as envisioned, the liquefaction plant will be located adjacent to a wastewater treatment plant and will take all the primary sludge generated by the wastewater plant--so in fact some credit is due since the sludge would otherwise have to be disposed of in some manner. Also, the cost of disposal will certainly rise in the future, as will petroleum prices. Therefore, the liquefaction process should become viable in future years even if it is not thought to be viable at current projected payback periods of 6-12 years.

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TABLE 8 PAYBACK PERIOD FOR SEVERAL SLUDGE LIQUEFACTION PROCESS OPTIONS

		FCI ($million)	$Million Manufacturing Costs	Sludge Disposal Credit	Oil/Asphalt Revenue	Total Revenue	Simple Payback (Years)
With Asphalt Recovery	With Dewatering Centrifuge	9.8	2.97	1.98	1.79	3.77	12
	Without Dewatering Centrifuge	7.8	2.68	1.98	1.79	3.77	7
Without Asphalt Recovery	With Dewatering Centrifuge	7.8	2.58	1.98	1.36	3.34	10
	Without Dewatering Centrifuge	5.8	2.29	1.98	1.36	3.34	6

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APPENDIX A

TEST RESULTS OF HONOLULU SLUDGE ASPHALT SAMPLES

PRELIMINARY TEST

Sample: HS#1 extracted and distilled

Date: April 14-16, 1980

Melting and Blending

The 100% HS melted near 35°C and became liquid near 60°C. Its viscosity-temperature curve might be steeper than for petroleum asphalt. The HS mixed and appeared to blend well with the AC-10 control petroleum asphalt. There was considerable odor from the hot HS sample. The 100% HS appeared to have some ductility.

Mixing with Hot Aggregate

Both the 100% HS and the 35-65% blend mixed well with hot aggregate. Because the 100% HS was more fluid, it mixed more quickly. Both mixes looked satisfactory after mixing, although the 100% HS mix sample looked more evenly coated. Considerable odor came from the 100% HS mix sample.

Oven Curing of Mixes

Both mixes were oven cured at 140°F for 16 hours. They looked about the same when removed from the oven, equally pliable. Odor was still present.

Water Exposure

Both mixes were covered with distilled water and placed in a 140°F oven for 10 hours. The containers were covered to inhibit evaporation. The water was decanted into beakers and examined. The 100% HS mix water was a medium amber color with some odor. The 35-65% blend mix water was a clear light amber with less odor. There were no noticeable signs of stripping, but the decanted water examination indicated the presence of dissolved material from the HS sample (especially the 100% HS sample). It is not known at present how detrimental this is from the points of view of reducing adhesive durability and secondary pollution effects.

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Heating and Compaction

Both mixes were dried, heated to 230°F and "compacted". The compaction was light: 20 tamps plus 13 psi static pressure. Both mixes compacted the same, although the 100% HS mix sample looked more uniform after compaction. Some odor was still present, especially in the 100% HS mix sample.

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Bending and Pulling

After compaction, the mixes were cooled in a 40°F refrigerator for 1 hour, and brought to room temperature for 1.5 hours. Each mix was bent in half to assess bending resistance and pulled apart to assess adhesion. The 100% HS mix had greater bending and tensile strength compared to the 35-65% HS blend mix. However, the 35-65% HS blend mix was slightly more ductile. No loss of interfacial adhesion between aggregate and binder was noticed in both the 100% HS and 35-65% HS blend mixes.

Overall Recommendation

There seems to be no severe telltale problems for the 100% HS#1 binder except for some odor and a little dissolving of a part of the HS material into water. The influence of the HS material will probably increase mix tensile-bending strength but may also impart more brittleness than with plain asphalt. It is not known if the HS material will age faster than petroleum asphalt. It is recommended that a larger sample of HS#1 synthetic asphalt be made and evaluated for compacted mix testing and asphalt behavior properties.

Note: The mix testing plus the asphalt testing will require 2.5 to 3.0 kg of HS#1.

PRELIMINARY TEST

Sample: HS#3 Sewage Sludge Asphalt

Date: June 3-5, 1980

Melting and Blending

The 100% HS#3 melted near 49°C and became liquid near 82°C. The HS#3 mixed awkwardly with the AC-10 control petroleum asphalt. The blend thickened up sort of like a gel. There was considerable odor from the hot HS#3 sample. The 100% HS#3 appeared to have ductility as noticed when a film (at room temperature) was placed on cardboard and bent 90°, but not as tacky as HS#1.

Mixing with Hot Aggregate

Both the 100% HS#3 and the 35-65% weight blend mixed with hot aggregate but not as well as with the HS#1 sample. Because the 100% HS#3 was more

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fluid, it mixed more quickly. Both mixes looked satisfactory after mixing although the 100% HS#3 mix sample was more evenly coated. Considerable odor came from the 100% HS#3 mix sample.

Oven Curing of Mixes

Both mixes were oven cured at 140°F (60°C) for 16 hours. They looked about the same when removed from the oven, equally pliable. Odor was still present.

Water Exposure

Both mixes were covered with distilled water and placed in a 140°F oven for 10 hours. The containers were covered to inhibit evaporation. The water was decanted into beakers and examined. The decanted 100% HS#3 mix water was a "muddy" olive brown color with sediment and some odor. The decanted 35-65% blend mix water was a less "muddy" olive brown with less sediment and odor. There were some signs of aggregate-binder dullness and the decanted water examination indicated the presence of suspended and dissolved material from the HS#3 sample (especially the 100% HS#3 sample). It is not known at present how detrimental this is from the points of view of reducing adhesive durability and secondary pollution effects.

Heating and Compaction

Both mixes were dried, heated to 230°F (100°C) and "compacted". The compaction was light: 20 tamps plus 13 (90 kPa) psi static pressure. Both mixes compacted the same. Some odor was still present, especially in the 100% HS#3 mix sample. The 100% HS#3 mix sample (as well as the 35-65% sample) appeared to regain an "asphalt mix" resemblance indicating that the leached material in the decanted water may not affect binding properties as severely as imagined.

Bending and Pulling

After compaction, the mixes were cooled in a 40°F (4.4°C) refrigerator for 1 hour, and brought to room temperature for 1.5 hours. Each mix was bent in half to assess bending resistance and pulled apart to assess adhesion. The 100% HS#3 mix had greater bending and tensile strength compared to the 35-65% HS#3 blend mix. However, the 35-65% HS#3 blend mix was slightly more ductile. No loss of interfacial adhesion between aggregate and binder was noticed in both the 100% HS#3 and 35-65% HS#3 blend mixes.

Overall Recommendation

Due to the very severe leaching of "impurities" in HS#3 and the awkward mixibility of the HS#3, it is concluded that the HS#1 sludge asphalt sample was superior to the HS#3 sample, and that a larger sample of HS#1 (approx. 2.5-3.0 kg) be made and evaluated for asphalt and compacted mix properties.

It should also be noted that all the sludge asphalt samples tested to date have the basics of good bonding properties and this is encouraging. Mix

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tests, as suggested for the HS#1 sample, would show whether the adhesion property is retained under the accelerated moisture conditioning. But at this time it is suggested that mix tests should not be pursued for the HS#3 sample.

PRELIMINARY TEST

Sample: HS#4 Sewage Sludge Asphalt

Date: June 18-20, 1980

Melting and Blending

The 100% HS#4 melted near 60°C and became liquid near 80°C. It appeared to have 5-10% "light oil" segregated in it. The sample was thoroughly mixed before proceeding further. The HS#4 mixed with the AC-10 control petroleum asphalt. The blend appeared uniform. There was some odor from the hot HS#4 sample. The 100% HS#4 appeared to have ductility and tackiness when a film (at room temperature) was placed on cardboard and bent 90°. The sample was not as brittle as the petroleum asphalt control, i.e. did not crack as easily (-12°C).

Mixing with Hot Aggregate

Both the 100% HS#4 and the 50-50% weight blend mixed with hot aggregate. Because the 100% HS#4 was more fluid, it mixed more quickly. Both mixes looked satisfactory after mixing, both mix samples were evenly coated. Some odor came from the mix samples.

Oven Curing of Mixes

Both mixes were oven cured at 140°F (60°C) for 16 hours. They looked about the same when removed from the oven, equally pliable. Slight odor was present.

Water Exposure

Both mixes were covered with distilled water and placed a 140°F oven for 10 hours. The containers were covered to inhibit evaporation. The leach water was decanted into beakers and examined. The decanted 100% HS#4 mix water was a "mud" olive brown color with sediment and some odor. The decanted 50-50% blend mix water was a less "muddy" olive brown with less sediment and odor. There were some signs of aggregate-binder dullness and the decanted water examination indicated the presence of suspended and dissolved material from the HS#4 sample (especially the 100% HS#4 sample).

Heating and Compaction

Both mixes were dried, heated to 230°F (110°C) and "compacted". The compaction was light: 20 tamps plus 13 (90 kPa) psi static pressure. Both

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mixes compacted the same. The 100% HS#4 mix sample (as well as the 50-50% sample) appeared to regain an "asphalt mix" resemblance.

Bending and Pulling

After compaction, the mixes were cooled in a 40°F (4.4°C) refrigerator for 1 hour, and brought to room temperature for 1.5 hours. Each mix was bent in half to assess bending resistance and pulled apart to assess adhesion. The 100% HS#4 mix had moderate bending and tensile strength, similar to the 50-50% HS#4 blend mix. Both blend mixes were ductile; the 100% HS#4 mix reflected greater ductility due to the inherent ductility of HS#4. No loss of interfacial adhesion between aggregate and binder was noticed in either the 100% or 50-50% HS#4 blend mixes.

Overall Recommendation

Due to the very severe leaching of "impurities" in HS#4, it is concluded that the HS#4 sludge asphalt sample is not as good as the HS#1 sample. The HS#4 sample, however is quite ductile and this is a good property.

PRELIMINARY TEST

Sample: HS#6 Sewage Sludge Asphalt

Date: June 25-27, 1980

Melting and Blending

The 100% HS#6 melted near 50°C and became liquid near 80°C. It had a strong odor. The HS#6 mixed with the AC-10 control petroleum asphalt. The blend appeared uniform. There was some odor from the hot HS#6 sample. The 100% HS#6 appeared to have putty-like consistency when a film (at room temperature) was placed on cardboard and bent 90°. At -12°C the sample seemed to be as brittle as the petroleum asphalt control. The putty-like behavior of the sample, rather than being tacky, indicated that this sample was not as good as some of the previous HS samples.

Mixing with Hot Aggregate

Both the 100% HS#6 and the 50-50% weight blend mixed with hot aggregate. Because the 100% HS#6 was more fluid, it mixed more quickly. Both mixes looked satisfactory after mixing, although the blend was more difficult to mix and coat the aggregate. Strong odor was present during mixing.

Oven Curing of Mixes

Both mixes were oven cured at 140°F (60°C) for 16 hours. They looked about the same when removed from the oven, equally pliable. Slight odor was present.

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Water Exposure

Both mixes were covered with distilled water and placed in a 140°F oven for 10 hours. The containers were covered to inhibit evaporation. The leach water was decanted into beakers and examined. The decanted 100% HS#6 mix water was a "mud" olive brown color with sediment and some odor. The decanted 50-50% blend mix water was slightly less "muddy" olive brown with much less sediment. There were some signs of aggregate-binder dullness and the decanted water examination indicated the presence of suspended and dissolved material from the HS#6 sample (especially the 100% HS#6 sample). This material and sediment appears to be from the HS sample itself rather than from any suspended clay-like particles in the aggregate.

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Heating and Compaction

Both mixes were dried, heated to 230°F (110°C) and "compacted". The compaction was light: 20 tamps plus 13 psi (90 kPa) static pressure. The 100% HS#6 mix sample (as well as the 50-50% sample) appeared to regain an "asphalt mix" resemblance in spite of the heavy leaching that had previously taken place in the water exposure.

Bending and Pulling

After compaction, the mixes were cooled to room temperature. Each mix was bent in half to assess bending resistance and pulled apart to assess adhesion. The 100% HS#6 mix had at least twice the bending and tensile strength of the 50-50% HS#6 blend mix. The 100% HS#6 mix reflected more brittleness during the test. No loss of interfacial adhesion between aggregate and binder was noticed in either the 100% or 50-50% HS#6 blend mixes.

Overall Recommendation

Due to the very severe leaching of "impurities" in HS#6, it is concluded that the HS#6 sludge asphalt sample is not as good as the HS#1 sample. The HS#6 sample behaves like a thermoplastic material with putty-like characteristics.

What is not known about the leaching problem is the compositional amount of the leachate. Does it consist of a fixed, small percentage (impurities) of the sludge asphalt? Or, does it consist of the larger amount of the sludge asphalt? In the former case, the leaching impurities could be removed prior to mixings leaving behind the basic binder. The bending and pulling test showed definite residual adhesion of the previously leached sludge asphalt; the residual binder had the outward appearance of asphalt. We will attempt to run a test to determine the amount of leachate (and of residual binder) after mixing with hot aggregate and exposing the loose mix to warm water under a few exposure time and agitation conditions. These results will be reported separately.

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Tests with several HS sludge asphalt samples show leaching of the samples in water. The leaching can be severe outwardly, imparting a muddy olive brown color to the water and producing a weakened asphalt concrete mixture in the presence of water. With the exception of sample HS-1, all the other HS samples showed significant leaching during the preliminary tests. Consequently additional tests were done to find out more about the leaching characteristics, and the results follow.

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HS Samples Used

Samples received to date that leached moderately severely to severely were subjected to further leaching tests. These samples were designated: HS-3, HS-4 and HS-6. Both HS 3 and HS-6 showed equally severe leaching and other characteristics; HS-4 showed moderately severe leaching with somewhat better ductility characteristics as compared to HS-3 and HS-6. There was not enough sample volume to provide for substantial testing at this stage. Therefore, different selected tests were performed on each sample.

HS-3 Tests

Two 6.5% permeable void compared mix test specimens were made using basaltic aggregate and HS-3. One specimen was vacuum saturated and accelerated conditioned in the presence of water. The leachate was olive light brown in color after 24 hr. in a 140°F water bath. The retained tensile splitting strength (55°F, .065 in. per min.) was only 12%. This is an unacceptable retained strength indicating that the warm water dissolved a portion of the HS-3 and that the water had a softening effect on the HS-3. When the wet specimen was dried, it regained some strength but it was weaker and had a tendency to crumble as compared to the dry specimen. It is apparent that water is detrimental to the adhesive nature of the HS-3.

HS-6 and HS-4 Tests

Loose mix samples of basaltic aggregate and HS-6 were made and subjected to static and to agitated action in warm water. The following are the results:

Warm Water Exposure Time, hr.	Leachate Removed, Percent of HS-6
8 (static)	8.4
16 (static)	9.5
32 (static)	10.5
8 (agitation)	25.8

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The results show about 9% of the HS-6 sample dissolved in warm water when it was mixed with aggregate and left in a static condition. When agitated and stirred, about 26% of the HS-6 sample dissolved -- giving about a maximum.

When these loose leached mixes were dried, reheated and compacted, the mix samples had regained strength somewhat but were relatively weak and crumbly. This indicates that some adhesive portions of the HS-6 may have dissolved in the water with the "impurities".

A loose mix sample of basaltic aggregate and HS-4 was made and subjected to agitated action in warm water. About 13% of the HS-4 sample dissolved -- giving the maximum amount. This was only one-half the maximum amount that leached from the HS-6, indicating that the HS-4 sample should have improved properties over the HS-6 sample. When the HS-4 loose leached mix sample was dried, rehated and compacted, it had regained some strength and its strength and ductility were better than the HS-6 samples, although not as satisfactory as a good petroleum asphalt sample.

Interim Summary

Severe leaching of the sludge asphalt - aggregate mixes means weakness and unsatisfactory behavior. If the sludge asphalt leaches 25% of its weight, its adhesion strength when wet reduces to about 12% of its preheated, dry strength. We suspect that only 1-2% max. leaching loss is tolerable after mixing with hot aggregate. Although not determined as of now, this implies that the HS-1 sample may be the only sample that is within the max. leaching loss limit after mixing with aggregate; preliminary tests on HS-1 indicated satisfactory behavior.

What is not known about the leachate is its composition and its binding property. If it consisted of only non-binder impurities, it could be washed out before hot aggregate mixing, making an acceptable binder for aggregate mixing.

Washing of Impurities from Sludge Asphalt

After the previously mentioned leaching tests, the only adequate amount of extra sludge asphalt sample remaining for a washing test was HS-6 (25 g).

The purpose of the washing test was to determine if a simple method could remove the approximately 26% of water "soluble" impurities from the HS-6 beforehand so that a more pure binder could be mixed with hot aggregate and evaluated. This would provide more adhesive film thickness on the aggregate surfaces and is much more desirable than the leaching after mixing which would remove or destroy a portion of the binder film thickness.

A simple method developed by the test staff that appeared successful consisted of placing the 25 g of HS-6 in a beaker in a 200°F (93°C) oven. When the HS-6 melted, 200°F (93°C) distilled water was added to the melted HS-6 in the beaker and stirred with a glass rod for a few minutes.

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Leaching began and continued in a static state (without mixing for an hour or two). The leachate was poured off and clean distilled water was added to the 200°F (93°C) HS-6 sample, stirred, and the additional leachate was poured off. This procedure was repeated a few more times until the poured- off distilled water was clear, indicating the full removal of impurities by this simple method. Cool water was added to the washed HS-6 in the beaker and the HS-6 "globbed", making for easy removal of the washed HS-6.

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The washed HS-6 was then "dried" by heating in the 200°F (93°C) oven until no visible signs of moisture were present, then heated to 300°F (149°C) and weighed. The weight loss was 27.2%, presumably due to the removal of impurities; it was close to the 26% observed during the aggregate mix leaching tests.

The washed, dried HS-6 at 300°F (93°C) was then mixed with hot aggregate for a preliminary test. (There wasn't enough sample to run a compacted mix test at this time).

PRELIMINARY TEST OF WASHED HS-6

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The washed, dried HS-6 mixed with hot aggregate was set in a pan covered with distilled water at 140°F (60°C) for 24 hr. The leaching was very slight, as expected, turning the water to a light yellow -- similar to the low amount observed in the HS-1 sample preliminary test. The water was poured off, the mix dried and heated to 230°F (110°C) and compacted. The bending and pulling strength of the washed HS-6 and aggregate mix was about equal to the preliminary test results of the mixleached sample of the original HS-6 and aggregate mix, but the washed HS-6 and aggregate mix exhibited more ductility and pliability. This indicates that the washing of impurities before aggregate mixing improved the binding characteristics of HS-6. How well it improved the binding characteristics, however, is not known without quantitative values determined by a compacted mix test series, but it is encouraging.

In order to determine if the bending and pulling of the washed HS-6 and aggregate mix had improved enough, qualitatively, to be equal to the petroleum asphalt control mix, a bending and pulling test was done with the Chevron AC-10 control asphalt and the hot aggregate (control mix). The control mix showed more plasticity and ductility than the washed HS-6 and aggregate mix with a rough qualitative rating of 2.5 better than the washed HS-6 and aggregate mix. The washed HS-6 and aggregate mix had good tensile strength, equal or better than the control mix, but it lacked the plasticity and ductility of the control mix.

Interim Conclusion

Leaching is a tell-tale of problems associated with reduced binding properties. When sewage sludge asphalt severely leaches from a compacted aggregate mix, the binding properties of the sludge asphalt are unsatisfactory. However, most of the "impurities" in the sludge asphalt can be washed out of the sludge asphalt before mixing with hot aggregate, producing an

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improved sludge asphalt binder with less aggregate mix leaching and with better plasticity and ductility. But the washed sludge asphalt still doesn't have the plasticity and ductility properties of the Chevron AC-10 control asphalt.

It therefore appears that a basically low leaching sludge asphalt such as HS-1 may be the best ranked so far to be competitive with the control asphalt. The quality of the sludge asphalt may have as much to do with the amount of cellulose present as with the extraction process itself.

PRELIMINARY TEST RESULTS OF HS-7

Washing

Melted HS-7 sample was washed in hot water by stirring in a beaker. The leachate was an olive brown color. The HS-7 sample lost approximately 30% (weight) after washing. The sample was then dried at 140°F (60°C).

Consistency

The washed HS-7 sample melted near 50°C and became liquid near 80°C. It had some characteristic odor. It was sticky and adhered well on cardboard when subjected to freezing temperature. At freezing temperature, the sample was brittle but no more so than petroleum asphalt. When exposed to room temperature, it regained its puttylike consistency much more rapidly than petroleum asphalt.

Mixing

A 50%-50% blend with the petroleum asphalt control was made as well as the 100% HS-7 "blend". There was some adverse blending reaction with the petroleum asphalt.

Both blends were mixed with hot aggregate and oven cured at 60°C for 16 hrs. When the loose mix was removed from the oven, the 50%-50% blend mix was duller and seemed to lack "life".

Water Exposure

Both blend mixes were covered with distilled water and placed in a 60°C oven for 10 hrs. The containers were covered to inhibit evaporation. The leach water was a light yellow color indicating that the main impurities were leached during the washing phase (step 1).

Heating and Compaction

Both blend mixes were dried, heated to 110°C and compacted. Both mixes showed adhesion and compactibility.

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Bending and Pulling

After compaction, the mixes were cooled to room temperature and then bent and pulled apart to assess adhesion. The 100% HS-7 mix was strong, (equally as strong as the previously good performing HS-1 mix). The 50%-50% blend mix was poor and crumbly, indicating the adverse reaction between HS-7 and petroleum asphalt, and therefore was declared unsatisfactory. No loss of interfacial adhesion was noticed with the 100% HS-7 mix and it was declared satisfactory at this stage.

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Compacted Mix Test Results of 100% HS-7

Compacted mix specimens made with 100% HS-7 and hot aggregate were subjected to dry and accelerated moisture conditioning (vacuum saturation followed by 0°C freezing and 60°C warm water soaking). Mechanical properties of tensile splitting strength and resilient modulus were obtained for dry and mixture conditioning to assess the mix's moisture sensitivity as compared to the petroleum asphalt control mix.

The following are the basic characteristics of the 100% HS-7 and petroleum asphalt compacted mixes prior to testing for tensile splitting and resilient modulus.

Petroleum Asphalt Control Mix:

Asphalt:	Chevron AC-10: pen. 82
Aggregate:	Crushed N. Idaho basalt; 100% pass 1/2 in. sieve, 66% pass No. 4 sieve, 27% pass No. 16 sieve, 13% pass No. 50 sieve, 6% pass No. 200 sieve
Asphalt Content:	6.0% (total mix wt. basis)
Permeable voids:	6.9% (by vacuum saturation)
Hveem stability:	35
Density:	151 lbs/ft^3 (2418.6 kg/m^3)

100% HS-7 Sewage Sludge Asphalt Mix:

Asphalt:	100% HS-7 sample (washed)
Aggregate:	Same as for petroleum asphalt control mix
Asphalt Content:	Same as for petroleum asphalt control mix

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Permeable voids: 6.0% (by vacuum saturation)

Density: 154 lbs/ft^3 (2460 kg/m^3)

The following are the results of the mechanical property tests under dry and moisture conditioning.

	Tensile Splitting Strength, psi* at 12.8°C			Resilient Modulus, psi* at 22.8 °C		
Type	Dry	Vac. Sat.	Accel. Cond.	Dry	Vac. Sat.	Accel. Cond.
Petroleum Asphalt Control Mix	99	97	78	203,000	210,000	155,000
100% HS-7 Sewage Sludge Asphalt Mix	81	--	50	254,00	243,000	254,000

*Note: Conversion: 1 psi - 6.895 kPa

The mechanical property magnitudes for the 100% HS-7 mix are close to those of the petroleum asphalt mix. There was more decrease in tensile strength than resilient modulus for the 100% HS-7 mix after accelerated conditions, but no indication of stripping was noted. The 100% HS-7 mix after accelerated conditioning was slightly dull looking as compared to the dry condition.

The following are the retained mechanical property percentages (based on dry property) after the mixes were subjected to moisture conditioning.

	Tensile Splitting Strength		Resilient Modulus	
Mix Type	Vac. Sat.	Accel. Cond.	Vac. Sat.	Accel. Cond.
Petroleum Asphalt Control Mix	98%	79%	103%	76%
100% HS-7 Sewage Sludge Asphalt Mix	--	62%	96%	100%

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The 100% HS-7 mix's retained tensile strength was lower than the petroleum asphalt mix, but its retained resilient modulus did not decrease after accelerated conditioning; the 62% retained tensile strength is lower than desired and places the 100% HS-7 mix in the "gray area" insofar as moisture sensitivity is concerned.

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CONCLUSIONS

1. The HS-7 sample after washing is the best Honolulu sludge asphalt sample tested to date. It behaves more like petroleum asphalt.

2. However, in contrast to petroleum asphalt, the HS-7 sample, similar to other sludge asphalts, is slightly more putty-like and has more consistency sensitivity to temperature change. It has good bonding behavior, however, and no stripping is noticed.

3. Hot aggregate mixes made with 100% HS-7 behave well and give tensile strength and modulus (stiffness) magnitudes close to the petroleum asphalt control mix.

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4. Moisture sensitivity of the 100% HS-7 compacted aggregate mix is slightly greater than the petroleum asphalt control mix, perhaps one performance category lower, for retained tensile strength. But its retained modulus (stiffness) is equal to or better than the control mix.

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Recommendation

The conditions in which the HS-7 sample was prepared and washed should form a base of data which could be slightly adjusted to make even a better sludge asphalt. It is recommended that the HS-7 formulation be used to develop the future work on sludge asphalt from the Honolulu source as part of a second phase effort to reach practical use.

PRELIMINARY TEST RESULTS OF HS-9

A small, unwashed sample was received for preliminary testing. (After melting, an oily fraction separated and later was decanted.) A portion was placed in thin film on cardboard and bent at room temperature. The sample broke, showing excessive brittleness.

Mixing

A 50%-50% blend with the petroleum asphalt control was made as well as the 100% HS-9 "blend". There was some adverse blending reaction with the petroleum asphalt.

Both blends were mixed with hot aggregate and oven cured at 60°C for 16 hrs. When the loose mix was removed from the oven, the 50%-50% blend mix was duller and seemed to lack "life".

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Water Exposure

Both blends were covered with distilled water and placed in a 60°C oven for 10 hrs. The containers were covered to inhibit evaporation. The leach water was a dark amber color indicating that main impurities were present in the sample.

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Heating and Compaction

Both blend mixes were dried, heated to 110°C and compacted. Both mixes showed adhesion and compactibility. The mixes lost heat rapidly and had to be continuously heated.

Bending and Pulling

After compaction, the mixes were cooled to room temperature and then bent and pulled apart to assess adhesion. The 100% HS-9 mix was crumbly and brittle. The 50%-50% blend mix was even more crumbly, indicating the adverse reaction between HS-9 and petroleum asphalt, and therefore was declared unsatisfactory. No loss of interfacial adhesion was noticed with the 100% HS-9 mix, but it was declared unsatisfactory at this stage because of impurities which probably produced a crumbly mix.

Compacted Mix Test Results of 100% HS-9 and 50%-50% HS-9

For compacted mix testing, an additional sample of HS-9 was received from Battelle that had been washed three times with distilled water at 100°C prior to distillation. Compacted mix specimens made with washed 100% HS-9 and with 50% HS-9 and 50% petroleum asphalt control and hot aggregate were subjected to dry and accelerated moisture conditioning (vacuum saturation followed by 0°C freezing and 60°C warm water soaking). Mechanical properties of tensile splitting strength and resilient modulus were obtained for dry and moisture conditioning to assess the mixes' moisture sensitivity as compared to the petroleum asphalt control mix.

The following are the basic characteristics of the HS-9 and petroleum asphalt compacted mixes prior to testing for tensile splitting and resilient modulus. Due to unusual rapid mix cooling with the HS-9 binder, the HS-9 mix compaction was more difficult and resulting voids were greater than the petroleum asphalt control mix.

Petroleum Asphalt Control Mix:

Asphalt: Chevron AC-10: pen. 82

Aggregate: Crushed N. Idaho basalt; 100% pass 1/2 in. sieve, 66% pass No. 4 sieve, 27% pass No. 16 sieve, 13% pass No. 50 sieve, 6% pass No. 200 sieve

Asphalt Content: 6.0% (total mix wt. basis)

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Permeable voids: 6.9% (by vacuum saturation)

Hveem stability: 35

Density: 151 lbs/ft^3 (2418.6 kg/m^3)

100% HS-7 Sewage Sludge Asphalt Mix:

Asphalt: 100% HS-9 sample, and 50% HS-9 and 50% petroleum asphalt control

Aggregate: Same as for petroleum asphalt control mix

Asphalt Content: Same as for petroleum asphalt control mix

Permeable voids: 8.8% (by vacuum saturation) for 100% HS-9 mix
11.4% (by vacuum saturation) for 50-50% HS-9 blend mix

Density: 150 lbs/ft^3 (2402.6 kg/m^3) for 100% HS-9 mix
145 lbs/ft^3 (2322.5 kg/m^3) for 50-50% HS-9 blend mix

The following are the results of the mechanical property tests under dry and moisture conditioning.

	Tensile Splitting Strength, psi* at 12.8°C			Resilient Modulus, psi* at 22.8°C		
Type	Dry	Vac. Sat.	Accel. Cond.	Dry	Vac. Sat.	Accel. Cond.
Petroleum Asphalt Control Mix	99	97	78	203,000	210,000	155,000
100% HS-9 Mix	183	--	20	1,898,000	1,259,000	102,000
50-50% HS-9 Mix	37	--	10	153,000	144,000	94,000

*Note: Conversion: 1 psi - 6.895 kPa

The mechanical properties for the 100% HS-9 mix are high, indicating strength but brittleness, whereas for the 50-50% HS-9 blend mix the properties are somewhat low. This contrast shows the reactive tendencies of mixing the HS-9 sewage asphalt binder with the petroleum asphalt control. The 100% HS-9 mix was hard, brittle with some cracks. The 50-50% HS-9 blend mix was crumbly.

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Mix Type	Tensile Splitting Strength		Resilient Modulus	
	Vac. Sat.	Accel. Cond.	Vac. Sat.	Accel. Cond.
Petroleum Asphalt Control Mix	98%	79%	103%	76%
100% HS-9 Mix	--	11%	66%	5%
50-50% HS-9 Mix	--	27%	94%	61%

The 100% HS-9 mix's retained tensile strength and resilient modulus after accelerated conditioning are too low and are unsatisfactory. The 50-50% HS_9 blend mix's retained tensile strength is too low and unsatisfactory; its retained resilient modulus is borderline "gray area".

CONCLUSIONS

The HS-9 sample extracted with lime is unsatisfactory as a petroleum asphalt substitute or blend, even if washed. The 100% HS-9 mix makes a brittle mixture when dry and it is highly water susceptible. It would not make a good pavement. The possible adverse reaction between HS-9 and petroleum asphalt in the 50-50% HS-9 blend mix produces a crumbly mix that is also water susceptible, giving a borderline-unsatisfactory performance.

RECOMMENDATION

The HS-9 sewage sludge asphalt sample is unsatisfactory as a petroleum asphalt substitute as a blend at the 50-50 level.

PRELIMINARY TEST RESULTS OF HS-10

A small sample was received for preliminary testing. It melted at 50°C and became liquid near 80°C. The sample retained heat and consistency better than the other HS samples. A portion was placed in thin film on cardboard and bent at room temperature; the sample was ductile. At freezing, the sample broke showing brittleness, but not unlike the petroleum asphalt control.

Mixing

A 50%-50% blend with the petroleum asphalt control was made as well as the 100% HS-10 "blend". There was some adverse blending reaction with the petroleum asphalt.

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Both blends were mixed with hot aggregate and oven cured at 60°C for 16 hrs. When the loose mix was removed from the oven, the 50%-50% blend mix was duller and seemed to lack "life".

Water Exposure

Both blends were covered with distilled water and placed in a 60°C oven for 10 hrs. The containers were covered to inhibit evaporation. The leach water was a clear brown color and a light yellow color for the 100% blend and for the 50% blend, respectively. There was no evidence of suspended solids in the leachate.

Heating and Compaction

Both blend mixes were dried, heated to 110°C and compacted. Both mixes showed adhesion and compactibility.

Bending and Pulling

After compaction, the mixes were cooled to room temperature and then bent and pulled apart to assess adhesion. The 100% HS-10 mix was strong and showed ductility. It showed good adhesion. The 50%-50% blend mix had low strength and was crumbly, indicating the adverse reaction between HS-10 and petroleum asphalt, and therefore was declared unsatisfactory.

Conclusions and Recommendation

The 100% HS-10 sample is potentially a good asphaltlike binder. Qualitatively it has good adhesion with aggregate and does not appear to have excessive moisture sensitivity in an aggregate mix. The HS-10 sample ranks with the HS-7 and HS-1 samples. The HS-10 sample seems to exhibit more heat retention properties than the other HS samples.

The adverse effects of mixing HS-10 with petroleum asphalt (50-50% blend) show up during the preliminary test, producing a weak, unsatisfactory mix.

It is recommended that the factors which produced HS-10 be further evaluated as a basis for making sewage sludge asphalt.

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APPENDIX B

EFFECT OF WATER-RELATED CONDITIONING ON INDIRECT TENSILE PROPERTIES OF COMPACTED BITUMINOUS MIXTURES

Scope

This method covers measurement of the change of diametral tensile strength and diametral (tensile) resilient modulus resulting from the effects of saturation and accelerated water conditioning of compacted bituminous mixtures. Internal water pressures in the mixtures are produced by vacuum saturation followed by a freeze and warm water soaking cycle. Numerical indices of retained indirect tensile properties are obtained by comparing the retained indirect tensile properties of saturated and accelerated water-conditioned laboratory specimens with the similar properties of dry specimens.

Apparatus

Two automatically controlled water baths for immersing the specimens. The baths are of sufficient size to permit total immersion of the test specimens. They are so designed and equipped as to permit accurate and uniform control of the immersion temperature. One bath is provided for bringing the immersed specimens to the temperature of 140 $\pm$ 3.6°F (60 $\pm$2°C) for the warm water soak part of the specimen conditioning. The second bath is provided for bringing the immersed specimens to either the selected test temperature of 55 $\pm$ 1.8°F (12.8° $\pm$ 1°C) or of 73 $\pm$ 1.8°F (22.8 $\pm$ 1°C) for the indirect tensile testing. The baths are constructed of or lined with stainless steel or other nonreactive material. The water in the baths is either distilled or otherwise treated to eliminate electrolytes and the baths are emptied, cleaned and refilled with fresh water for each series of tests.

One automatically controlled freezer for freezing the specimens. The freezer is of sufficient size to permit total containment of the test specimens. It is so designed and equipped as to permit accurate and uniform control of its air temperature. The freezer is required for bringing the specimens to the selected temperature of -0.4 $\pm$ 3.6°F (-18 $\pm$ 2°C) for the freeze portion of specimen accelerated conditioning.

One vacuum pump with capacity to pull at least 26 in (66 cm) of mercury for the water saturation of the test specimens. Accessory equipment includes: Pyrex or equivalent vacuum jars of at least 6 in. (15 cm)

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diameter and 8 in. (20 cm) high with smooth fired edges, a donut shaped gasket made of rubber-type sponge, a stiff metal round plate greater than 6 in. (15 cm) diameter with suitable vacuum hose receptacle and hole bored through the plate thickness, vacuum hose attached to receptable fitting and vacuum pump, and a 6 in. (15 cm) diameter screen-type or highly porous specimen spacer seat approximately 0.25 in. (1 cm) high.

A compressive testing machine as described in accordance with Method D 1074, but having the controlled deformation rate capability of 0.065 in per min (0.165 cm per min).

Mark III or Mark IV Resilient Modulus Apparatus manufactured by Retsina Co., El Cerrito, CA 94530, or equivalent.

A balance and a room-temperature water bath with suitable accessory equipment for weighing the test specimens in air and in water (saturated specimens only) in order to determine their densities, the amount of absorption, and permeable voids. This apparatus is similar to that required for Method D2726, Bulk Specific Gravity of Compacted Bituminous Mixtures Using Saturated Surface-Dry Specimens.

A supply of plastic film for wrapping and heavy-duty leak-proof plastic bags to wrap and enclose the saturated specimens for preventing moisture loss during handling and freezing.

Several metal jars of at least 4 in. (10.2 cm) diameter and at least 6 in. (15 cm) high for bringing dry specimens to test temperature without water intrusion into the dry specimens in the water bath.

Test Specimens

At least nine, duplicate 4 in. (102 mm) diameter by 2.5 in. (63.5 mm) high cylindrical test specimens of the same mixture are made for each test. The procedures described in either: Method D1559, Test for Resistance to Plastic Flow of Bituminous Mixtures Using Marshall Apparatus, or Method D1561, Test for Compaction of Test Specimens of Bituminous Mixtures by Means of California Keading Compactor, or Method D3387, Test for Compaction and Shear Properties of Bituminous Mixtures by Means of the U.S. Corps of Engineers Gyratory Testing Machine, are followed in preparing the loose mixtures and in molding and curing the test specimens. The same aggregate and asphalt (and additive, if necessary) source and grade expected for the road application are contained in the mixtures. It is suggested that the loose mixtures be cured in pans for 15h at 140°F (60°C) prior to further heating to compaction temperature. Specimens are compacted to the most probably permeable void content.

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Grouping, Vacuum Saturation, and Determination of Bulk Density and Permeable Voids of Test Specimens

Allow each set of nine test specimens to cool at room temperature for at least 24 hours after completion of specimen fabrication and curing described in Methods: D1559, D1561, D3387. Label each specimen with waterproof identification and obtain the dry weight of each specimen to the nearest 0.1 g.

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Randomly select a subset, I, of three specimens from the set of nine test specimens. Maintain subset I specimens in a dry condition. Place subset I specimens in metallic jars and then place the jars in a water bath at the selected mechanical test temperature (see Note 1) of 55 ± 1.8°F (12.8 ± 1°C) or 73 ± 1.8°F (22.8 ±1°C) for 5 h maintaining the top lip of the jars above the water level of the bath. Place an insulating stuffing in the top of the jars, making contact with the top specimen's surface and with the jar walls, then proceed with the mechanical testing of subset I as described in sections 6-9.

The six remaining test specimens are vacuum saturated as follows. Place a porous spacer seat on the bottom of a vacuum jar and then place two or more of the specimens, depending on jar height, horizontally in the jar using another porous spacer seat between the specimens. Put distilled water, or water treated to eliminate electrolytes, at 73°F (22.8°C) in the jar to about 1 in (2.5 cm) above the upper specimen's surface. Place a dampened donut gasket and a stiff metallic plate on top of the jar. Attach a vacuum hose from the plate receptacle to the vacuum pump. Apply a vacuum of 26 in (66 cm) of mercury to the jars for a duration of 30 min, gently agitating the jar wall. Remove the vacuum and leave the six specimens submerged in the jars at atmospheric pressure for 30 minutes.

Remove each of the six specimens from the vacuum jars, quickly surface dry the specimens by towel blotting and weigh immediately in air and then weigh submerged in room-temperature water at approximately 73°F (22.8°C). Immediately after weighing each submerged specimen, return the specimens to the water-filled vacuum jars and submerge each specimen under the water at atmospheric pressure.

Calculate the bulk density and permeable voids of each of the six vacuum saturated test specimens as follows:

$$\text{Bulk density} = \frac{AD}{B-C},$$

$$\text{Permeable voids, \%} = \frac{100\ (B-A)}{B-C},$$ where

A = weight of dry specimen in air, g,

B = weight of surface-dry (blotted) vacuum saturated specimen in air, g,

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C = weight of vacuum saturated specimen submerged in water, g, and

D = density of water at 73°F (22.8°C), g/cc

Sort and assign each of the six vacuum saturated test specimens into two subset, II and III, consisting of three specimens each so that the average permeable voids (or average bulk density) is essentially the same in each subset. Immerse subset II specimens into a water bath at the selected mechanical test temperature of 55 ± 1.8°F (12.8 ± 1°C) or 73 ± 1.8°F (22.8 ± 1°C) for 3 h and then proceed with the mechanical testing of this subset described in sections 6-9. Condition the subset III specimens by using the procedure described in section 5.

Accelerated Conditioning Procedure

Maintain specimen surface dampness and internal saturation, and wrap tightly each of the three specimens of subset III with two layers of plastic film using masking tape to hold the wrapping if necessary. Place each wrapped specimen into a leak-proof plastic bag, and seal the bag with a tie or tape.

Immerse each of the three individually wrapped and bagged specimens of subset III into an air bath freezer for 15 h at -0.4 ± 3.6°F (-18 ± 2°C). (If this step is completed at 5 pm, specimens can be removed from the freezer at 8 a.m. the following day).

Remove the three wrapped and bagged specimens of subset III from the freezer and immerse them immediately into a water bath at 140 ± 3.6°F (60° ± 2°C) for 24 hr. (After 1/2-1 h of immersion, when specimen surface thaw takes place, carefully remove the bag and wrapping from the specimens and reimmerse the specimens in the water bath).

Carefully remove the three unwrapped specimens of subset III from the water bath, immerse the specimens in a water bath at the selected mechanical test temperature of 55 ± 1.8°F (12.8 ± 1°C) or73 ± 1.8°F (22.8 ± 1°C) for 3 h, and then proceed with the mechanical testing of this subset as described in sections 6-9.

Note 1. Refer to section 6 for information on the selection of mechanical test temperature.

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Selection of Mechanical Test Temperature

The selection of the mechanical test temperature for the nine specimen set is based on the type of mechanical test desired for measurement of the effects of the water-related conditioning. Diametral (tensile) resilient modulus may be performed at either 55 ± 1.8°F (12.8 ± 1°C) or 73 ± 1.8°F (22.8 ± 1°C). Diametral tensile strength is performed at 55 ± 1.8°F (12.8 ± 1°C). If low to moderate stresses are applied to the specimens in the diametral (tensile) resilient modulus test, then this test can be considered nondestructive and the same specimens can be also tested using the diametral tensile strength test, therefore providing additional mechanical properties data. If this is to be done, then specimens at 73°F (22.8°C) or 55°F (12.8°C) must be reimmersed in the water bath at 55F(12.8C) for 3h or 1/2h, respectively, prior to the diametral tensile strength testing.

Specimen Handling in the Mechanical Testing Procedures

Each specimen subset is tested rapidly following the completion of their respective test-temperature water-bath soak times as prescribed in section 4.2 for subset I, section 4.6 for subset II, and section 5.4 for subset III.

Remove a subset specimen from the water bath at the test temperature, surface dry by blotting with a towel (necessary for specimens from subsets II and III), measure and record the specimen height (thickness) and identification, and place the specimen wth circular ends vertical (specimen on edge) into the appropriate mechanical loading device. Test one specimen at a time, leaving the remaining untested specimens in the water bath. Proceed with testing as rapidly as possible since the mechanical testing will expose the specimen to air temperature which may be different than the test temperature. Test the specimens by either one or both of the procedures described in sections 8 and 9.

Test and Calculation Procedure for Diametral (Tensile) Modulus

Place the transducers of the Resilient Modulus Apparatus on the specimen at test temperature and proceed quickly with diametral loading at 0.1 sec. load duration time, following the procedures described in the instruction manual provided by the manufacturer. Record load and horizontal deformation. Rotate the specimen 90° and repeat.

Calculate the specimen's diametral resilient modulus for each of the two 90° rotations as follows:

$$M_R = \frac{P\ (+\ 0.2734)}{L},$$ where

M_R = diametral resilient modulus, psi (k Pa),

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P = load magnitude applied to specimen, lb (dN),

= Poissons ratio of specimen (use 0.35 unless measured specifically),

0.2734 = dimensionless strain integration constant for 4 in. (10.2 cm) diameter specimens,

L = thickness of specimen, in. (cm), and

= horizontal deformation magnitude of specimen, in. (cm).

The average of the two 90° resilient modulus values is calculated for this specimen and test temperature. Return specimen to water bath if a diametral tensile strength test is also to be performed on the same specimen.

Repeat by testing the two remaining specimens in the subset, and calculate the overall average diametral resilient modulus for the subset of three specimens.

Repeat procedure and calculations described in sections 8.1-8.1 for the remaining two subsets of three specimens.

Proceed to section 10, calculation.

Test and Calculation Procedure for Diametral Tensile Strength

Place and center a subset specimen at test temperature under the flat loading head of the compression test machine, and proceed quickly with diametral loading at a vertical deformation rate of 0.065 in. per min (0.165 cm per min). (The specimen is placed on its edge without support blocks or loading strips). Record the maximum compressive load. Immediately decrease load to zero, remove specimen and measure specimen edge or side flattening to nearest 0.1 in. (0.25 cm). This can be accomplished easily by stroking the top flattened edge (side) with a piece of chalk held lengthwise and horizontally to delineate the flattened width, and then using a scale to measure the average maximum width of the flattened edge. Record this width. Replace the specimen in the compression test machine with its original orientation (flattened edges top and bottom) and redeform the specimen at 0.065 in. per min (0.165 cm per min) until a definitive vertical crack appears and opens. Decrease load to zero, remove specimen and slowly pull apart the two sides of the specimen at the crack. The internal surface may then be observed for stripping, and recorded qualitatively.

Calculate the specimen's diametral tensile strength as follows:

$$S_t = \frac{S_{10}\, P}{10{,}000\, L}, \quad \text{where}$$

S_t = diametral tensile strength, psi (k Pa),

S_{10} = maximum tensile stress, psi (k Pa), obtained by calculating: $1591 + 437a - 1889\, a^2 + 2854\, a^3 - 2474\, a^4 + 885\, a^5$, where a = flattening width, inc., based on a 4 in. (10.2 cm) diameter solid cylinder loaded at 10,000 lb (22 kg) per inch (cm) thickness (see note 2),

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P = maximum compressive load on specimen, lb (N),

10,000 = load constant: 10,000 lb per in of thickness (17,512 N per cm of thickness), and

L = thickness of specimen, in (cm).

Repeat by testing the two remaining specimens in the subset, and calculate the overall average diametral tensile strength for the subset of three specimens.

Repeat procedure and calculations described in sections 9.1-9.4 for the remaining two subsets of three specimens each.

Proceed to section 10, Calculation.

Note 2 To calculate S_{10} in SI units, first calculate S_{10} in U.S. Customary units of psi using the polynomial constants as shown, with a in inches, then convert psi to k Pa using 1 psi = 6.895 k Pa.

Calculation

Calculate the numerical indices of the effects of vacuum saturation and accelerated conditioning as the ratios of the mechanical properties of subsets II and III to the mechanical properties of subset I for the specified test temperature as follows:

$$M_R R_1 = \frac{M_R (II)}{M_R (I)} \quad \text{and} \quad M_R R_2 = \frac{M_R (III)}{M_R (I)}, \quad \text{where}$$

$M_R R_1$ = diametral resilient modulus ratio of saturation,

$M_R R_2$ = diametral resilient modulus ratio of accelerated conditioning,

$M_R (I)$ = average diametral resilient modulus of specimen subset I, psi (k Pa),

$M_R (II)$ = average diametral resilient modulus of specimen subset II, psi (k Pa), and

$M_R (III)$ = average diametral resilient modulus of specimen subset III, psi (k Pa).

$$TSR_1 = \frac{S_t (II)}{S_t (I)} \quad \text{and} \quad TSR_2 = \frac{S_t (III)}{S_t (I)}, \quad \text{where}$$

TSR_1 = diametral tensile strength ratio of saturation,

TSR_2 = diametral tensile strength ratio of acclerated conditioning,

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S_t (I) = average diametral tensile strength of specimen subset I, psi (k Pa),

S_t (II) = average diametral tensile strength of specimen subset II, psi (k Pa), and

S_t (III) = average diametral tensile strength of specimen subset III, psi (k Pa).

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Ratios will be reported to the nearest hundredth.

Ratios may be interpreted as follows. M_RR_1 and TSR_1 are related to short-term pavement performance, eg. 2 yr. M_RR_2 and TSR_2 are related to long-term pavement performance, eg. 8 yr. Low ratios are associated with the compacted mixture's inability to resist moisture effects.

Precision

Single-Operator Precision - The single operator standard deviation has been found to be 11% for M_RR and 10% for TSR (see Note 3). Therefore, results of two properly conducted tests by the same operator on the same material should not differ by more than 28% for M_RR and 31% for TSR.

Multilaboratory Precision - This value is currently not available. Data are currently being obtained from cooperative testing among seven state and federal highway agencies.

Note 3 These numbers represent, respectively, the (IS) and (D 2S) limits as described in ASTM Recommended Practice C 670, for Preparing Precision Statements for Test Methods for Construction Materials.

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TABLE C-1 COMPOUNDS IDENTIFIED BY GC/MS ANALYSIS OF SLUDGE SAMPLES

Sample Description	Analysis Conditions	Compound Identification	MW	Probability	Peak Number
Headspace of HS-7, taken within eight hours of its removal from autoclave. The sludge was stored in plastic pails with tight-fitting plastic lids.	Headspace injection volume 20 cc. column was 25 m OV-1 glass WCOT capillary. Temperature program 60-100°C at 2°/min	Methyl butanol	88	T	1
		furan	68	P	5
		acetone	58	P	6
		2-methyl furan	82	H	7
		pentan-2-one	86	P	9
		thiophene	84	H	10
		toluene	92	H	11
		2,3-dimethyl disulphide	94	H	12
		2-methyl thiophene	98	H	13
		ethyl benzene	106	H	14
		methyl thiophene	98	P	15
		2,5-dimethyl thiophene	112	P	16
		2-ethyl thiophene	112	P	17
		n-methyl pyrrole	81	H	18
		3-ethyl thiophene	112	T	19
		2,4-dimethyl thiophene	112	T	20
		1-ethyl pyrrole	95	P	21
		3-methyl cyclopentanone	98	P	23
		cyclooctatetraene	104	T	24
Headspace of sample several weeks after it had been removed from the autoclave. Sample was stored in plastic bucket with tight-fitting lid.	Headspace injection volume 25 cc. column 25 m OV-17 glass WCOT. Isothermal at 70°C	Allyl vinyl ether	84	T	1
		2-pyrone	96	P	3
		acetone	58	H	4
		pentan-2-one	86	H	6
		toluene	92	P	7
		2,3-dimethyl disulphide	94	H	8
		3-methyl thiophene	98	P	9
		dihydroxy acetone	90	T	10
		ethyl benzene	106	H	11

TABLE C-1 (Continued)

Sample Description	Analysis Conditions	Compound Identification	MW	Probability	Peak Number
		2-methyl thiophene	98	H	12
		N-methyl pyrrole	81	H	14
		1-ethyl pyrrole	95	P	15
		pentane thiol	104	T	16
Steam-volatile sample HS-1, stored in glass vial with polyethylene cap.	Injection size 1.4 ul. Column was 25m cp wax glass WCOT capillary. Temperature programming 40-240°C /min	Toluene	92	H	1
		ethylbenzene	106	H	3
		dimethyl benzene isomer	106	H	5
		2,3-dimethyl thiophene	112	T	9
		cyclooctatetraene	104	T	11
		2-methoxyaniline	123	T	15
		2-ethyl-1-hexanol	130	H	21
		3-acetyl-2,4-dimethyl pyrrole	137	T	35
		2-(n-methyl 1-carbamoyl)-benzal-dehyde	179	T	42
		phenol or phenylmethyl-carbamate	94 or 159	T	46
		ethyl phenol isomer	122	T	49
		p-cresol	108	P	50
		ethyl phenol isomer	122	T	53
		n-octadecanol	270	P	62
		indole	117	H	63
		methyl indole	131	H	64
		ethyl indole	145	H	66

* H = Highly probable, P = Probable, T = Tentative

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TABLE C-2 GC/MS ANALYSIS OF VOLATILE COMPOUNDS

Compound Name	MW	Confidence Level of Identification* Headspace 1	Headspace 2	Distillate
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Acetone	58	P	H	
Allyl vinyl ether	84		T	
Pentan-2-one	86	P	H	
Methyl butanol	88	T		
Dihydroxy acetone	90		T	
2,3-dimethyl disulphide	94	H	H	
3-methyl cyclopentanone	98	P		
Cyclooctatetraene	104	T		T
Pentane thiol	104		T	
2-ethyl-1-hexanol	130			H
n-octadecanol	270			P
HETEROCYCLICS				
Furan	68	P		
2-methyl furan	82	H		
n-methyl pyrrole	81	H	H	
1-ethyl pyrrole	137			T
thiophene	84	H		
2-methyl thiophene	98	H	H	
Methyl thiophene	98	P	P	
two dimethyl thiophene	112	T		T
isomers		P		
2-ethyl thiophene	112	P		
3-ethyl thiophene	112	T		
2-pyrone	96	P		

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TABLE C-2 (Continued)

Compound Name	MW	Confidence Level of Identification* Headspace 1	Headspace 2	Distillate
indole	117			H
methyl indole	131			H
ethyl indole	145			H
AROMATICS				
toluene	92	H	P	H
phenol or phenylmethyl-carbamate	94 151			T
two dimethyl benzene isomers	106			H
ethyl benzene	106	H	H	H
p-cresol	108			P
two ethyl phenol isomers	122			T
Ethyl benzene	106	H	H	H
p-cresol	108			P
two ethyl phenol isomers	122			T
2-methoxy aniline	123			T
2-(n-methylcarbamoyl)-benzaldehyde	179			T

* H = Highly probable, P = Probable, T = Tentative.

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TABLE C-3 ELEMENTAL ANALYSIS: C,H,N,O,S (%)

Element	Petroleum Asphalt AC-10	HS-4	HS-5	HS-6	HS-9	HS-10	HS-9*	HS-10*	Leachate HS-6	Char cake HS-10
%C	87.03	76.84	69.37	72.50	73.07	78.31	75.38	78.12	27.13	
	86.79	75.98	68.70	72.56	73.91	77.90	75.97	77.89	58.90	24.91
									59.21	25.06
Average	86.91	76.41	69.04	72.53	73.49	78.11	75.68	78.01	59.06	25.70
%H	11.23	9.35	9.52	9.71	9.37	9.70	11.53	11.87	8.88	3.42
	10.61	8.99	9.67	9.72	9.68	9.85	12.04	11.65	9.00	3.38
										3.29
Average	10.92	9.17	9.58	9.72	9.53	9.78	11.79	11.76	8.94	3.36
%N	0.38	2.33	4.61	4.73	4.45	2.48	2.77	2.41	2.77	1.21
	0.43	2.21	4.58	4.68	4.55	3.59	2.32	2.50	2.94	0.75
										0.80
Average	0.41	2.27	4.60	4.71	4.50	3.04	2.55	2.46	2.86	0.92
%O	1.40	8.41	11.42	10.22	10.64	8.12	6.58	6.50	20.18	10.44
	0.77	6.06	14.57	7.74	4.42	5.23	11.47	2.46	16.81	9.01
Average	1.09	7.24	13.00	8.98	7.53	6.68	9.03	4.48	18.50	9.73
%S		0.77 ±0.03	0.57 ±0.03	0.64 ±0.03	1.05 ±0.04	0.82 ±0.03	1.11 ±0.06	0.75 ±0.04	0.87 ±0.04	

*Replicate determinations

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